STABILITY STUDIES OF QUARTZ CRYSTALS

FOR SATELLITES

by

RICHARD B. BELSER

and

W. H. HICKLIN

Project A--508

Engineering Experiment Station
Georgia Institute of Technology
Atlanta, Georgia
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CONTENTS

GEORGIA INSTITUTE OF TECHNOLOGY

ENGINEERING EXPERIMENT STATION
ATLANTA 13, GEORGIA

5 August 1960

Frequency Control Division
Electronic Components Research and Development Laboratories
Fort Monmouth, New Jersey

Attention: Dr. E. A. Gerber

Subject: Progress Letter No. 1
Contract No. DA-36-039-SC-85363
Georgia Tech Project No. A-508
Period: 10 June 1960 to 1 August 1960

Dear Sir:

The purpose of this project is to investigate the effects of time, temperature and corpuscular radiation on the frequency stability of 100 Mc AT-cut quartz crystal units. Target objectives are maximum aging rates of $\pm$ 0.3 ppm/yr. with ultimate objectives of frequency changes not greater than $\pm$ 0.1 ppm/yr.

The 100 Mc measuring apparatus and ovens for resonator storage, partially constructed under Contract No. DA-36-039-SC-78905, were completed and placed in operation. The measuring apparatus required little additional adjustment; two of the ovens, on the other hand, were modified to operate at 60°C and to cycle from 0°C to 60°C, respectively, and the third was completed to operate at 0°C. These ovens are presently undergoing calibration tests and final temperature adjustment.

A separate room has been established to contain an Atomichron frequency standard and the three quartz crystal oscillator standards, type O-76U. The latter are being supplied with a standby battery power supply to take over immediately by means of an automatic switching system in case of failure of the local AC-supply. The crystal oscillators will be monitored against the Atomichron and against signals transmitted from frequency standard sources such as WWV or the Boulder, Colorado station of the National Bureau of Standards. From the described equipment a standard signal will be supplied to any desired site within the laboratory by means of a coaxial cable. The Atomichron is on hand and will be installed within the next 10 days.

Plans for the month of August are to complete the preliminary equipment needs and to solve the preliminary measurement problems. In addition, groups of resonators for storage and frequency measurement at 60°C will be fabricated. It is planned to begin with 5th overtone units base-plated with Al. The effects of frequency adjustment with overcoats of Al and of Ag will be defined.

The personnel employed and the time devoted to the project by each during the month of July are shown below.

	Hours
Richard B. Belser, Project Director	35
Douglas W. Robertson, Research Engineer	25
Walter Hicklin, Assistant Research Engineer	168
James O. Darnell, Research Assistant	160
Don Dawson, Student Assistant	168
W. B. Warren, Jr., Research Engineer	25

Mr. Warren has been devoting time primarily to the installation of the Atomichron and the O-76U oscillators in order to provide a standard frequency signal of the required accuracy for the intended measurements. This installation is expected to be completed within the next thirty days.

Respectfully submitted,

Richard B. Belser
Project Director

Approved:

for Vernon Crawford
Head, Physics Branch
Physical Sciences Division

GEORGIA INSTITUTE OF TECHNOLOGY
ENGINEERING EXPERIMENT STATION
ATLANTA 13, GEORGIA

September 6, 1960

Frequency Control Division
Electronic Components Research and Development Laboratories
Fort Monmouth, New Jersey

Attention: Dr. E. A. Gerber

Subject: Progress Letter No. 2
Contract No. DA-36-039-SC-85363
Georgia Tech Project No. A-508
Period: 1 August 1960 to 1 September 1960

Dear Sir:

The purpose of this project is to investigate the effects of time, temperature variations and corpuscular radiation on the frequency stability of 100 Mc AT-cut quartz crystal units. Target objectives are maximum aging rates of $\pm$ 0.3 ppm/yr with an ultimate objective of frequency changes not greater than $\pm$ 0.1 ppm/yr.

Mr. R. B. Belser, of this laboratory, visited USASRDL on 31 August 1960 and discussed the progress and future plans of the project with Messers Gutwein, Bernstein and Mulvihill.

The Atomichron primary frequency standard (on loan from the Air Force for a few months) has been installed and is in operation. The three Western Electric Oscillators, Type O-76U, have likewise been installed and are being monitored against the Atomichron. Frequency changes of the order of one part in 10^9 per day are being experienced by the O-76U Oscillators.

The vacuum equipment, used in crystal plating, has been modified to include a liquid nitrogen cold trap introduced directly into the plating chamber. A modification to include a gettering action of the chamber during plating has also been made. This consists of a separate filament and power supply, protected by a series of baffles. A getter material such as barium, misch metal or titanium is evaporated from this filament prior to the plating of the quartz plates. By these aids a pressure below 3×10^{-7} has been obtained during plating operations. Improvements in these methods are expected to extend the useful pumping range of the system somewhat below the pressures obtained to date.

Construction of a final plating chamber and of a vacuum baking chamber utilizing the techniques described above is now under way. Thus all operations *in vacuo* will be conducted at pressures in the pressure range of 10^{-7} mm of mercury.

Fifteen 100 Mc resonators have been fabricated. These consisted of six seventh-overtone units and nine ninth-overtone units base plated with aluminum and not overcoated. Difficulty was experienced with the first group in obtaining properly sealed glass envelopes and only one of these units was considered hermetically sealed. This unit experienced a frequency drop of three parts in 10^7 in 19 days at 60°C. R_s values of the units ranged from 50 ohms to values too high to operate. The high values were ascribed in part to poor finish of the edges of the particular blanks.

A second group of nine units was placed in the oven on 23 August. These units, in 14 days, have experienced negative frequency shifts of 1.5 to 6 parts in 10^7 at 60°C. R_s values of these units ranged from 40 to 60 ohms.

New glass envelopes and stems are being ordered and an additional supply of crystals with polished edges will be obtained.

Plans for the month of September are to continue fabrication of 100 Mc resonators utilizing a base coat of aluminum or silver only. Subsequently, units will be fabricated utilizing Al + Al and Al + Ag.

The persons employed and the time devoted by each during the month of August are shown below.

	Hours
Richard B. Belser, Project Director	64
Walter H. Hicklin, Assistant Research Engr.	160
James O. Darnell, Research Assistant	160
William D. Dawson, Student Assistant	160

Respectfully submitted,

Richard B. Belser
Project Director

Approved:

Vernon Crawford
Head, Physics Branch
Physical Sciences Division

GEORGIA INSTITUTE OF TECHNOLOGY

ENGINEERING EXPERIMENT STATION

ATLANTA 13, GEORGIA

November 23, 1960

Frequency Control Division
Electronic Components Research and Development Laboratories
Fort Monmouth, New Jersey

Attention: Mr. E. A. Gerber

Subject: Progress Letter No. 3
Contract No. DA 36-039-SC-85363
Georgia Tech Project A-508
Period 1 October 1960 to 1 November 1960

Dear Sir:

The purpose of this project is to investigate the effects of time, temperature variations and corpuscular radiation on the frequency stability of 100 mc AT-cut quartz crystal units. Target objectives are maximum aging rates of ± 0.3 ppm/yr with an ultimate objective of frequency changes not greater than ± 0.1 ppm/yr.

During the period since the last report the 0°C, 60°C and 0 to 60°C cycling ovens have been completed and set in operation. Work has continued on the frequency measurement system in a search for the cause of inaccuracy which has prevented measurement accuracy of better than ± 1 part in 10^7. For some units the apparent measurement accuracy is ± 2 parts in 10^8, the objective, but an uncertainty exists that confuses the measurements. The units of higher impedance, and of higher overtones, have given the greatest stability. Currently efforts are being made to trace the source of error to impedance variations due to temperature change of the coaxial line, line position change, and contact resistance.

The 18 KC signal from Station NBA of Balboa, Canal Zone, has proved of sufficient strength and phase stability for calibration of the local O-76-U frequency standards. Measurements of this signal have been shown to be accurate in Atlanta, Georgia, to within 6 parts in 10^{10} by checks against the Atomichron, available here currently on a temporary, loan basis.

Seventeen additional 100 Mc resonators, making a total of 54 during the life of the project, have been fabricated. These consisted of eight fifth overtone units plated with Al only and 9 fifth overtone units plated with Al + Ag. Only 10 of the 17 units were of a quality warranting further testing. The high mortality in yield was due in part to the questionable quality of the quartz plates used.

The best aging results over a period of 45 days have been obtained from ninth overtone units plated with silver only. Unit D-9-2 shows only 9 parts in 10^8 change during this period. Units plated with Al only have shown several parts in 10^7 drift during the same interval. Graphs showing the frequency data for selected units are being forwarded to Dr. G. K. Gutwein under separate cover.

The first quarterly report has been completed and draft copies will be forwarded in a few days. More complete details of the experimental work will be found in this report.

Additional resonators of the fifth, seventh and ninth overtones will be plated with Al or Ag and overcoated to frequency with the respective metal. Aging measurements of these and previously fabricated units will be made. Efforts in improving measurement accuracy will be continued.

Respectfully submitted,

Richard S. Delser
Project Director

Approved:

Vernon Crawford
Head, Physics Branch
Physical Sciences Division

cc: Addee (5)

GEORGIA INSTITUTE OF TECHNOLOGY

ENGINEERING EXPERIMENT STATION

ATLANTA 13, GEORGIA

December 5, 1960

Frequency Control Division
Electronic Components Research and Development Laboratories
Fort Monmouth, New Jersey

Attention: Mr. E. A. Gerber

Subject: Progress Letter No. 4
Contract No. DA-36-039-sc-85363
Georgia Tech Project A-500
Period: 1 November 1960 to 1 December 1960

Dear Sir:

The purpose of this project is to investigate the effects of time, temperature variations and corpuscular radiation on the frequency stability of 100 mc AT-cut quartz crystal resonators. Target objectives are maximum aging rates of ± 0.3 ppm/year with an ultimate objective of frequency changes not greater than ± 0.1 ppm/year.

Work has been continued on efforts to improve the present measuring techniques for measurements at 100 mc frequency. Alternatives existing have been examined. These are: (1) an improved coaxial line from the bridge to the controlled temperature oven; (2) moving the bridge from its present position at the CI meter TS-15 to the base of the oven in which measurements are being made; (3) the adoption of a passive measurement system in which the impedance of the crystal is measured over a range of frequencies supplied from a stable external oscillator. Preliminary measurements have shown that (2) is unlikely to be feasible and that (3) may be a fruitful approach accurate to one part in 10^8. The improvements of (1) are being made with the hope that this step may suffice to bring measurements immediately to an accuracy of ± 3 or 4 parts in 10^8 or better.

Sixteen additional resonators have been fabricated. Only seven of the 16 were operable. These were ninth overtone units, base plated with silver only, and show stable performance over the test period since fabrication. Resistance values were in the range 75 to 90 ohms.

One unit of a preceding group (unit B-9-5) was intentionally punctured. Whereas the unit had exhibited stable behavior previously it now began to drop roughly 200 cycles per day. The remainder of this group have continued stable and one unit (B-9-4) has shown variations of only ± 5 parts in 10^8 from its initial frequency in 60 days.

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The first quarterly report was completed and approval copies were forwarded on 22 November 1960.

A second Crystal Impedance Meter, TS-15, is badly needed by the project in order to carry out measurements of the R_s and the Q of crystals now being fabricated. The present bridge frequency measurement system completely ties up the project's CI meter TS-15 and no other equipment capable of making the measurements is available to the project.

Plans for the succeeding month are to make the outlined improvements in frequency measurement and to proceed with the planned fabrication of additional resonators. Measurements of fabricated units will be continued. Quantities of fabricated units have been reduced by the necessity for spending more time than anticipated on measurement apparatus and technique development. Studies of radiation effects are also being delayed pending more reliable aging data measurements.

Respectfully submitted,

Richard D. Belser
Project Director

Approved:

Original signed by Vernon Crawford

Vernon Crawford
Head, Physics Branch
Physical Sciences Division

cc: Addee (5)

GEORGIA INSTITUTE OF TECHNOLOGY
ENGINEERING EXPERIMENT STATION
ATLANTA 13, GEORGIA

5 January 1961

Frequency Control Division
Electronic Components Research and Development Laboratories
Fort Monmouth, New Jersey

Attention: Dr. E. A. Gerber

Subject: Progress Letter No. 5
Contract No. DA-36-039-sc-85363
Georgia Tech Project A-508
Period 1 December 1960 to 1 January 1961

Dear Sir:

The purpose of this project is to investigate the effects of time, temperature variations and corpuscular radiation on the frequency stability of 100 mc AT-cut quartz crystal units. Target objectives are maximum aging rates of ± 0.3 ppm/yr with an ultimate objective of frequency changes not greater than ± 0.1 ppm/yr.

During the month of December measurements of 100 mc resonators previously fabricated have been continued and 8 additional silver plated fifth overtone units have been fabricated.

Work has been continued on the search for improved frequency measurement techniques along the lines previously discussed.

The screen voltage applied to the electron tubes in the CI meter TSM-15 has been stabilized by supplying it directly from a 45-volt battery, an associated voltage divider and helipot. This increases the oscillator stability by eliminating the effects of rapid small variations in line voltage to which the screen voltage was previously subject.

Two additional VHF bridges have been constructed. One was built to accommodate four coaxial lines. Two of the lines were connected to a resistor and two to the crystal. However, the hoped for impedance balance was not successful since during a test run there was no certainty that the circuit was crystal controlled.

The second bridge was build so that it may be used with the crystal operating as an active resonator or as a passive element. In initial tests where the bridge was inserted in the RF output of the CI Meter

(Experimental Model) a lack of precise resettability was observed. However, a more thorough investigation of the potentialities of this system is now underway.

The use of teflon coaxial lines and General Radio line stretchers was examined with a view to obtaining improvement over the polyethylene coaxial connector lines previously used. The teflon lines proved to be so stiff as to impair uniform reconnection. The line stretchers were resettable only to about 5 parts in 10^7.

The use of a new set of polyethylene lines terminated with BNC terminators No. UG-88 C/U has given some improvement in accurate frequency measurement after reconnection. However, the substitution of this new pair of lines caused frequency shifts of somewhat under 1 ppm for all resonators on aging measurement.

Further studies of the bridge frequency measurement method have revealed that the VHF bridge itself is somewhat temperature sensitive and an oven is now being constructed to control the bridge temperature at 5°C above the ambient; the ambient, in turn, is to be held more nearly constant by installation of an additional individual room air conditioner.

Plans for the next month are to continue the search for improved frequency measurement accuracy and to fabricate additional resonators. Measurements of resonators now on hand will be continued. Progress Report No. 2 will be completed and forwarded.

Respectfully submitted,

Richard B. Belser
Project Director

Approved:

Vernon Crawford
Head, Physics Branch
Physical Sciences Division

GEORGIA INSTITUTE OF TECHNOLOGY
ENGINEERING EXPERIMENT STATION
ATLANTA 13, GEORGIA

5 February 1961

Frequency Control Division
Electronic Components Research and Development Laboratories
Fort Monmouth, New Jersey

Attention: Dr. E. A. Gerber

Subject: Progress Letter No. 6
Contract No. DA-36-039-SC-85[illegible]
Georgia Tech Project A-508
Period: 1 January 1961 to 1 February 1961

Dear Sir:

The purpose of this project is to investigate the effects of time, temperature variations and corpuscular radiation on the frequency stability of 100 mc quartz crystal units. Target objectives are maximum aging rates of ± [illegible] ppm/year with an ultimate objective of frequency changes not greater than ± 0.1 ppm/year.

During the month of January measurements of 100 mc resonators have been continued and 20 additional units have been fabricated. These consisted of 10 fifth overtone units plated with silver only and ten plated with copper only. Of the twenty units only eight would successfully operate since the blanks plated were of an initial supply of 0.375 diameter blanks. This diameter appears unsuitable for best results in this overtone and frequency range.

The Second Quarterly Report, for the period 1 October 1960 to 1 January 1961, was completed and forwarded during the month.

In the Quarterly Report two bridge-frequency-measurement systems were described; one was based on a system modified from the one developed here by D. J. Robertson; the second was based on a system suggested by B. [illegible] of this project. The latter, which was described in the report, is isolated from the CI Meter by a ferrite transformer and operated with one side of the crystal to ground. This system has proved less sensitive to lead-handling effects than the first and has now been adopted as the principal measurement device. The measuring equipment has been adapted for its use, and the Experimental Crystal Impedance Meter No. 2, Serial No. 1, built by D. J. Robertson,

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is being used as the oscillator for the circuit. The drive level of the CI Meter TS-15, previously used for this purpose had been limited by modification and was found to be unsatisfactory for use with the new measuring system.

Thus far the newly adopted bridge system appears to permit measurements to an accuracy of 1 part in 10^8.

During the month the frequency control project was given an additional room. The space was utilized to establish a fabrication division and a measurement division in separate rooms. The move was completed partly in anticipation of the additional contract which requires additional facilities and partly to permit the exercise of better control over both fabrication and measurement.

Plans for the next month are to complete evaluation of the new measurement system and to expand fabrication rates and aging measurements. Facilities for radiation exposure of units will be prepared.

Respectfully submitted,

Richard B. Belser
Project Director

Approved:

Original Signed By Vernon Crawford

V. Crawford
Head, Physics Branch
Physical Sciences Division

GEORGIA INSTITUTE OF TECHNOLOGY

ENGINEERING EXPERIMENT STATION

ATLANTA 13, GEORGIA

[illegible] March 1961

Frequency Control Division
Electronic Components Research and Development Laboratories
Fort Monmouth, New Jersey

Attention: Dr. E. [illegible] Gerber

Subject: Progress Letter No. 7
Contract No. D[illegible]-SC-[illegible]
Georgia Tech Project No. [illegible]
Period: 1 February 1961 to 1 March 1961

The purpose of this project is to investigate the effects of time, temperature variations and corpuscular radiation on the frequency stability of 100 mc quartz crystal units. Target objectives are maximum aging rates of ± [illegible] ppm/year with an ultimate objective of frequency changes not greater than ± 0.1 ppm/year.

During the month of February the move into larger quarters, begun in January, was completed in order to take on an additional aging study work load. All the vacuum systems and fabrication equipment were moved into one laboratory and all the aging and measuring equipment into a room immediately adjacent. This arrangement promotes maximum precision in both fabrication and measurement.

Concurrently with the adoption of the grounded terminal bridge it was necessary to modify the measuring equipment and ovens for operation with it. In addition, an automatic gain control circuit for stabilization of the oscillator screen voltage was designed and built. The latter is now ready for installation. The measuring equipment, so modified, gives promise of fulfilling an accuracy requirement of approximately ± 2 parts in 10^9 as a minimum, even for the fifth overtone units which have displayed erratic behavior in previous measurements.

Because of the considerable time devoted to the move and the revision of measuring equipment, only ten resonators were fabricated during the month. These were fifth overtone units, base plated with silver. Of these the yield was only three crystals satisfactory for aging measurements.

Arrangements were made with the Georgia Tech Radio Isotopes Laboratory for radiation exposure of resonators for which suitable aging data had been obtained. A [illegible] curie cesium 137 source is available for radiation. The radiation from this source is so intense that glass becomes discolored and brittle in an exposure of one hour. As a prelude to these experiments a complete spectrographic analysis of all materials used in the resonator mount is being conducted.

The Van der Graaf source for 1 MEV protons is currently undergoing repair but is expected to be in operation within 30 days. Contacts are also to be made with the Oak Ridge Nuclear Laboratories for possible exposure of some resonators to the proton beam of a cyclotron at higher energies.

Plans for the month of March are to concentrate on fabrication and measurement. Initial experiments on radiation exposure of the resonators will be conducted.

Respectfully submitted,

Richard B. Belser
Project Director

Approved:

V. Crawford
Head, Physics Branch
Physical Sciences Division

GEORGIA INSTITUTE OF TECHNOLOGY

ENGINEERING EXPERIMENT STATION
ATLANTA 13, GEORGIA

April 5, 1961

Frequency Control Division
Electronic Components Research and Development Laboratories
Fort Monmouth, New Jersey

Attention: Dr. E. A. Gerber

Subject: Progress Letter No. 8
Contract No. DA-36-039-SC-85363
Georgia Tech Project No. A-508
Period: 1 March 1961 to 1 April 1961

Dear Sir:

The purpose of this project is to investigate the effects of time, temperature variations and corpuscular radiation on the frequency stability of 100 mc quartz crystal units. Target objectives are maximum aging rates of ± 0.3 ppm/year with an ultimate objective of frequency changes not greater than ± 0.1 ppm/year.

During the month of March frequency measurements of crystals stored at 60°C and at 0°C have been continued. The new method of driving the VHF bridge and the AGC system, described in the last monthly summary, have proven to be suitable for the measurement precision required.

Two groups of 5th overtone units plated with evaporated silver were fabricated. The first group of ten (group B-5) was base plated only. The yield of acceptable units was 100%. The average series resistance of the group after vacuum baking and sealing was 25 ohms. The highest resistance was 39 ohms, the lowest 17 ohms. The second group (H-5) was plated to frequency with evaporated silver. The yield for this group was only 20% due primarily to poor plate-back technique which caused the crystal resistance to increase. A modification made on the frequency plating mask has apparently corrected the trouble and a second silver plus silver group has been completed.

Data have been accumulated to indicate that the glass stems* now being used are subject to small leaks at the glass to metal seals where the leads enter the stems. The stems formerly obtained from the General Electric Company gave no such trouble and steps are being taken to procure new stems and envelopes from the General Electric Company at Nela Park, Cleveland, Ohio.

Nine crystal units were removed from the 60°C oven and sent to the radiation laboratory for exposure to radiation from a 12,000 curie cesium source. The following table summarizes the result of the experiment to date.

TABLE I

Parameters of Quartz Resonators Exposed for Various Times to Radiation from 12,000 Curie Cesium Source

Unit Designation*	Plating	Over-tone	Exposure Time (Minutes)	ΔF** (Cycles)	ΔR_s** (Ohms)	30 Days Aging Prior to Exposure*** ($\times 10^{-8}$)	10 Days Aging After Exposure*** ($\times 10^{-8}$)
A-9-2	Ev Al	9th	10	- 545	+ 8	± 0.0	+74
A-9-3	Ev Al	9th	10	- 340	+18	± 0.0	+39
A-9-4	Ev Al	9th	20	- 527	+ 7	+ 8.0	+34
A-9-6	Ev Al	9th	20	- 323	+ 5	+ 7.0	+17
A-9-7	Ev Al	9th	30	-1048	+16	- 4.0	+53
A-9-9	Ev Al	9th	30	-1097	+24	+ 3.0	+70
B-9-2	Ev Ag	9th	10	- 280	+10	- 4.0	+26
B-9-4	Ev Ag	9th	20	- 730	---	± 0.0	+43
B-9-6	Ev Ag	9th	30	- 737	---	+ 4.0	+19

* All mounted in evacuated glass bulbs.

** Calculated from measurements at 60°C before and after exposure.

*** Where aging has been two cycles or less, the aging rate is assumed to be essentially zero.

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*Made by WRW Scientific Glass Company, Inc. (Bliley Electric Company Drawing K-475-M.)

Evaluation of the data obtained during radiation studies will be completed in time for the Third Quarterly Report due 31 April 1961. A visible darkening of the glass envelope occurred during the radiation; the degree of darkening was dependent on the time of exposure. It is worthy of note that the radiation each unit will receive will be affected by the thickness of the specific glass envelope and that the uniformity of the envelopes as to thickness may not be very good.

Plans for the month of April include fabrication of additional crystal units with emphasis placed on 7th overtone units plated with evaporated silver. Additional specimens will be subjected to radiation and measurement of the effect of the exposure on the frequency and aging of the units. The Third Quarterly Report will be prepared.

Respectfully submitted,

Richard B. Belser
Project Director

Approved:

Original Signed By Vernon Crawford

Vernon Crawford
Head, Physics Branch
Physical Sciences Division

cc: Addee (5)

bcc: A. L. Bennett
V. Crawford
R. B. Belser
Library (2)
Surplus

GEORGIA INSTITUTE OF TECHNOLOGY
ENGINEERING EXPERIMENT STATION
ATLANTA 13, GEORGIA

3 May 1961

Frequency Control Division
Electronic Components Research and Development Laboratories
Fort Monmouth, New Jersey

Attention: Dr. E. A. Gerber

Subject: Progress Letter No. 9
Contract No. DA-36-039-SC-85363
Georgia Tech Project A-508
Period: 1 April 1961 to 1 May 1961

Dear Sir:

The purpose of this project is to investigate the effects of time, temperature variations and corpuscular radiation on the frequency stability of 100 mc quartz crystal resonators. Target objectives are a maximum frequency change of ± 0.3 ppm/year with an ultimate objective of a frequency change not greater than ± 0.1 ppm/year.

During the month of April frequency measurements of crystal units stored at 0°C and 60°C have been continued. All equipment performed well and no major repairs were necessary.

Four groups of crystal units were completed. Pertinent details of each group are tabulated below.

TABLE I

Resonators Fabricated During April 1961

Group	No Units	Over-tone Order	Base Plate	Final Plate	Yield	R_s (ohms)	Bonding Cement	Remarks
J-5	10	5	Ag	Ag	8	25	Hanovia No. 2	
K-5	10	5	Ag	-	8	25	duPont 5504 A	R_s averaged for six units
B-7	10	7	Ag	-	10	47	duPont 5504 A	" " " eight units
C-7	10	7	Ag	Ag	8	52	duPont 5504 A	

Total units = 40 Total yield = 85%

The units which were exposed to the maximum dosage of gamma radiation from the 12,000 curie source for periods of from 10 to 30 minutes have been measured for over 30 days. All of the units appear to be approaching stable operation after initial positive aging at a rate of about 5 parts in 10^8 per day. The plateau on the frequency vs time curve for a storage temperature of 60°C indicates an average change of +6.0 parts in 10^7 for the 9 units. The glass bulbs which were darkened by the gamma radiation, on examination after 30 days, appeared normal. Units given a reduced gamma dosage regained stability in about 5 days after a total positive aging of about 5 parts in 10^8. Results of the 1 Mev proton radiation from the Van de Graaff are inconclusive.

Seven units, the aging rate of which had been established at 0°C, were temperature cycled between 0°C and 60°C at a rate of 5°C/hour for nine days. The results are given on the following table.

TABLE II

Stability Measurement of Resonators Temperature Cycled once Daily over Range 0°C to 60°C

Unit	Overtone Order	Base Plate	R_s (ohms)	Bonding Cement	Crystal Mount	ΔF* (ohms)	ΔR_s* (ohms)
A-5-1	5	Al	~10	Hanovia No. 2	0.006" Springs	+1	±0.0
A-5-3	5	Al	33	"	"	+2	±0.0
A-5-9	5	Al	~10	"	"	+1	±0.0
A-5-10	5	Al	20	"	"	-3	±0.0
C-5-2	5	Al	39	"	Tab Clips**	+195	+2.0
C-5-3	5	Al	30	"	"	+28	±0.0
C-5-5	5	Al	~10	"	"	+99	±0.0

* Measured at 0°C after cycling for 9 days.

** Tab clips were obtained from Diebel Tool and Die Co., Chicago, Ill.

The data exhibited above indicate that more study is required on crystal mounting structures. The 0.006 inch spring clip seems to be superior to the tab clip from the standpoint of stability during temperature cycling. The spring clip mount, however, lacks the mechanical rigidity to withstand the effects of shock, vibration and acceleration as well as the tab clip. Subsequent measurement at 0°C on the cycled units over a three day period indicate that the units mounted in spring clips have maintained their initial aging rate while those in tab clips are aging downward at a greater rate of about 5 parts in 10^8 per day.

Research areas of current interest that will be conducted during the succeeding months of May and June, within the funds available, are as follows.

1. A continuation of the investigation of silver-plated, 7th mode units now underway.
2. Studies of the effect of temperature cycling on frequency with emphasis placed upon the role of the crystal mount structure.
3. A continuation of the effects of radiation upon frequency and aging rate.
4. The effects of crystal drive level on aging.

A decision as to whether the work will be extended after June 30, 1961 is needed in order that proper planning in the use of personnel and equipment may be made. The solution to the measurement problem, which was a project within itself, will allow concentration of all effort during any additional work directly on fabrication and aging studies under the stipulated conditions of temperature and irradiation.

Sincerely yours,

Richard B. Belser

Approved:

Vernon Crawford
Head, Physics Branch
Physical Sciences Division

GEORGIA INSTITUTE OF TECHNOLOGY
ENGINEERING EXPERIMENT STATION
ATLANTA 13, GEORGIA

5 June 1961

Frequency Control Division
Electronic Components Research and Development Laboratories
Fort Monmouth, N. J.

Attention: Progress Letter No. 10
Contract No. DA-36-039-SC-85363
Georgia Tech Project A-508
Period 1 May 1961 to 1 June 1961

Dear Sir:

The purpose of this project is to investigate the effects of time, temperature variations and corpuscular radiation on the frequency stability of 100 mc quartz crystal resonators. Target objectives are a maximum frequency change of ± 0.3 ppm/year with an ultimate objective of a frequency change not greater than ± 0.1 ppm/year.

During the month of May frequency measurements of crystal units stored at 0°C and 60°C have been continued; measurements of frequency changes occurring for units exposed to temperature cycling over the range 0° to 60°C daily and on exposure to gamma radiation and proton bombardment have been conducted. A summary report of work to date was presented by Mr. R. B. Belser and Mr. W. H. Hicklin at the 14th Annual Frequency Control Symposium at Atlantic City, N. J. on 31 May 1961; and the paper submitted for the proceedings contains a detailed account of work conducted up through approximately 20 May 1961.

Only ten additional resonators were fabricated during the month. These were 7th overtone units base plated with silver and not overcoated, group D-7. These particular units were stored at 85°C in order to observe the action of accelerated aging, considering the short remaining time for which the project is funded.

Frequency measurements have been continued on approximately 60 resonators in addition; approximately one-half of these were stored at 0°C and one-half at 60°C. In addition seven units were subjected to temperature cycling over the daily range 0° to 60°C for a period of 7 days and 16 units were subjected to exposure to the cesium beam or Van de Graaff irradiators.

In general results observed have confirmed those discussed in some detail in Quarterly Report No. 3; in addition these results, were reported at the recent symposium.

In short, the measurement problem has been resolved so that measurements to an accuracy of ± 2 parts in 10^8 can be repeatedly made. Short term aging measurements (60 days) and some long term ones (over 180 days) have indicated that aging of less than 0.3 ppm per year can be obtained from selected units. Better results have been obtained with silver than with aluminum plating although aluminum plated resonators will also remain stable after an initial shift of a few parts in 10^7. Stresses, developed during thermal cycling gave undesirable frequency shifts for some units, presumably due to mounting rigidity.

High intensity gamma radiation, similar to that developed as a result of 600 kev electron collisions with a satellite or a resonator container, caused negative frequency shifts. These were as high as 10 ppm in a few minutes at high intensities (1.6×10^6 rad/hr) but dropped to 1 or 2 parts in 10^7 per day at intensities near these reported in the Van Allen belt. After irradiation, partial recovery in a slow upward frequency drift occurred; a second irradiation dose resulted in a smaller shift than the first.

Proton bombardment on the other hand (1 Mev protons) resulted in only small shifts. Four of seven units examined shifted upward 3 to 5 parts in 10^7, one exhibited no change, one shifted negatively and the container of one was cracked by the beam. An exposure of one unit to the beam for 480 sec at a rate of 1.4×10^4 rad/sec resulted in an upward shift of 1.3 ppm. This was the maximum exposure time and the maximum shift observed for resonators under proton bombardment.

The program for the next month envisions a continuation of studies now underway and expansion of the radiation effect studies to observe the effects of radiation saturation. Continuous monitoring of the frequency shift versus time for units inserted into the radiation field at various intensity levels is expected to give the desired information. Tabulation of data for the Final Report will be commenced.

Since notice of extension of the Project has not been received and no surplus funds exist beyond those required for completion of the report, research will be discontinued on or about the 30th of June pending the receipt of additional funds.

Respectfully submitted,

/
Richard B. Belser
Project Director

Approved:

V. Crawford
Head, Physics Branch
Physical Sciences Division

GEORGIA INSTITUTE OF TECHNOLOGY
ENGINEERING EXPERIMENT STATION
ATLANTA 13, GEORGIA

5 July 1961

Frequency Control Division
Electronic Components Research and Development Laboratories
Fort Monmouth, New Jersey

Attention: Dr. E. A. Gerber

Subject: Progress Letter No. 11
Contract No. DA-36-039-sc-85363
Georgia Tech Project A-508
Period 1 June 1961 - 1 July 1961

Dear Sir:

The purpose of this project is to investigate the effects of time, temperature variations and corpuscular radiation on the frequency stability of 100 mc quartz resonators. Target objectives are a maximum frequency change of $\pm$ 0.3 ppm/year with an ultimate objective of a frequency change not greater than $\pm$ 0.1 ppm/year.

During the month of June frequency measurements of resonator units stored at 0°C and 60°C have been continued; measurements of frequency changes occurring for units exposed to temperature cycling over the range 0° to 60°C daily and on exposure to gamma radiation have been conducted.

Nine additional resonators, three each from the 5th, 7th, and 9th overtone groups, have been exposed to maximum intensity radiation in the well of the Ce 137 source. Intensities were of the order of 1.4×10^6 rad/hr. Measurements were made, at the fundamental frequency only, at intervals up to approximately 22 hours. The resonator was retrieved from the well and allowed to cool to the air conditioned room temperature for each measurement (about five minutes). The temperature in the well was approximately 10°C above room temperature. The measurements taken on these resonators are shown in Table I attached; a graph showing a typical behavior pattern (Figure 1) is also inclosed.

It is worthy of note that the largest negative frequency shift occurs in the first few minutes and that thereafter the negative drift slope is much smaller or non-existent. After several hours the frequencies begin to rise again. For all nine resonators a plus value was indicated for the final reading of 17 to 22 hours. This was of the magnitude of +1 to +18 ppm. In general, the 7th and 9th overtone resonators exhibited smaller shifts than the 5th overtone resonators. R_s values exhibited relatively small changes.

No measurements have yet been made on post saturation dose frequency drifts. This appears to be a highly desirable measurement since it would reveal whether radiation pretreatment of resonators would be a condition for maximum stability under subsequent radiation.

No additional resonators were fabricated during June since aging measurements for an appreciable time could not be made on them prior to expiration of the funded period of the project.

Work on the summary report was begun.

Plans for the month of July are to shift the primary emphasis of the work to Contract No. DA-36-039-sc-87407 (Georgia Tech project A-552) since sufficient funds do not remain in this project to support work other than the preparation of the final report.

It is our belief that work should be continued on Contract No. DA-36-039-sc-85363; however, unless funds for its continuation are made available in the near future, it will be necessary for us to shift both personnel and equipment to other projects.

Respectfully submitted,

Richard B. Belser
Project Director

RBB/var

Addressee, 5

bcc: A. L. Bennett
R. B. Belser, 2
Library, 2
Surplus

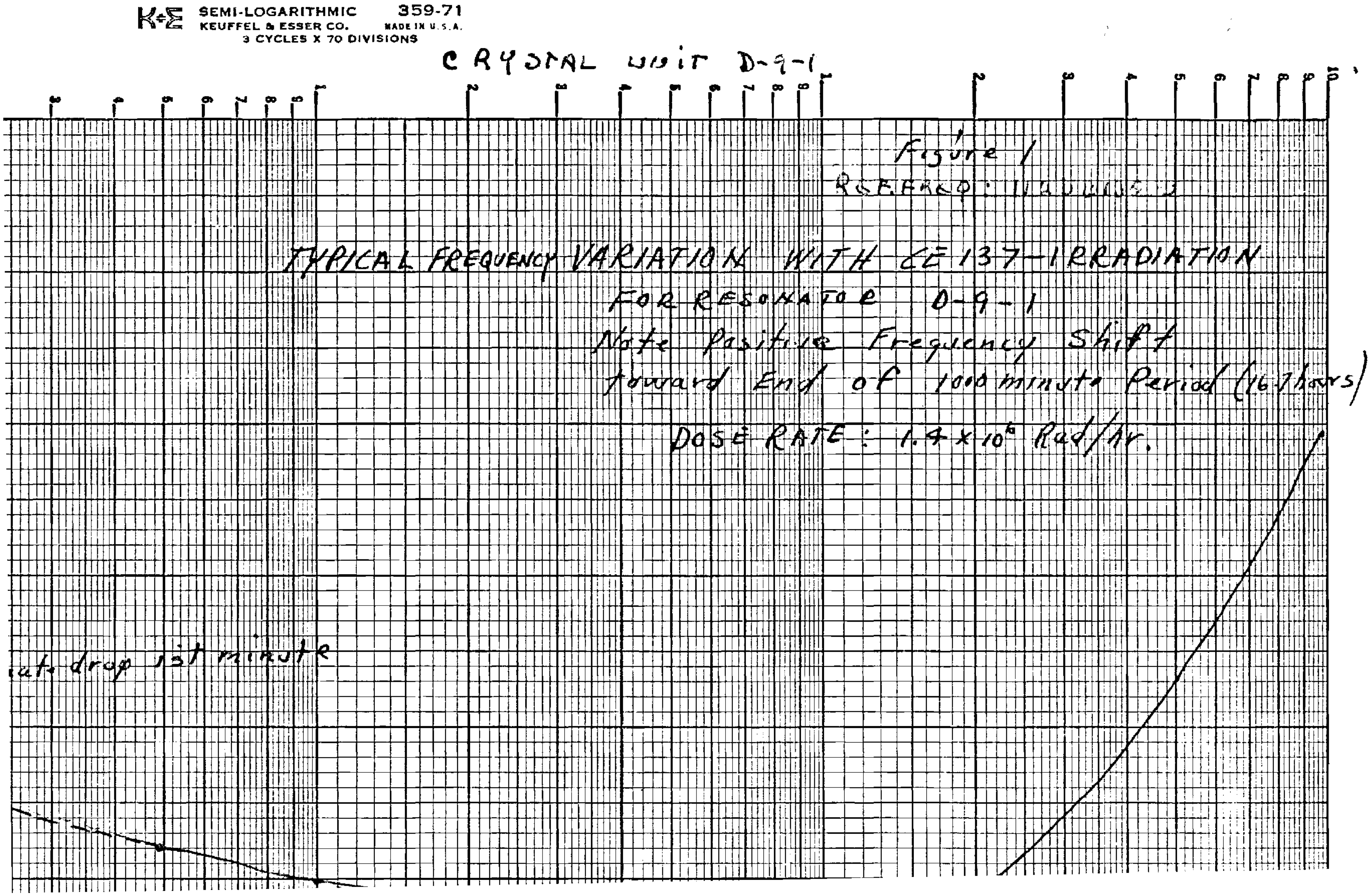

K+E SEMI-LOGARITHMIC 359-71
KEUFFEL & ESSER CO. MADE IN U.S.A.
3 CYCLES X 70 DIVISIONS
CRYSTAL UNIT D-9-1
Figure 1
REF.FREQ:
TYPICAL FREQUENCY VARIATION WITH CE 137 - IRRADIATION
FOR RESONATOR D-9-1
Note Positive Frequency Shift
toward End of 1000 minute Period (16.7 hours)
DOSE RATE: 1.4 x 10^6 Rad/hr.
...ate drop 1st minute

TABLE I

EFFECTS OF Ce-137 IRRADIATION ON QUARTZ RESONATORS

(Studies Subsequent to 1 June 1961)

9th Overtone Units (Operated at Fundamental)

Frequency and R_s Values at Various Times

Unit D-9-1-Ag

Time of Irradiation	0	5'	10'	20'	50'	90'	973'
Frequency Shift (cycles)	Initial Value 11, 236, 130	-58	-69	-79	-76	-81	+70
ppm	0	-5.2	-6.1	-7.1	-6.8	-7.2	+6.2
R_s Values(ohms)	4.0	4.0	4.5	-	4.0	4.0	4.5
D-9-8-Ag (Frq)	Initial Value 11, 229, 981	-50	-53	-59	-55	-60	+23
ppm	0	-4.5	-4.8	-5.3	-4.9	-5.3	+2.1
R_s (ohms)	5.5	5.5	6.0	6.0	-	-	6.0
D-9-7-Ag (Frq)	Initial Value 11, 227, 071	-52	-68	-82	-104	-108	+69
ppm	0	-4.6	-6.0	-7.3	-9.3	-9.6	+6.1
R_s	4.0	4.5	5.0	5.0	5.0	-	5.0

7th Overtone Units

Frequencies and R_s Values at Various Times

B-7-2-Ag

Time (min)	0	5	10	15	30	167	287	1292
Frequency Shift (Cycles)	Initial Value 14, 353, 890	-89	-88	-88	-87	-87	-68	+13
ppm	0	-612	-6.2	-6.2	-6.1	-6.1	-4.8	+0.9
R_s (ohms)	5.0	5.0	6.0	6.0	5.0	5.0	5.0	5.0
B-7-6-Ag	Initial Value 14, 341, 690	-58	-97	-99	-134	-155	-162	+120
ppm	0	-4.1	-6.8	-7.0	-9.4	-10.9	-11.3	+8.4
R_s (ohms)	5.0	5.0	5.5	5.5	5.5	5.0	-	5.0
C-7-2-Ag	14, 264, 906	-38	-74	-75	-109	-153	-144	+254
ppm	0	-2.7	-5.2	-5.3	-7.6	-10.6	-10.1	+17.7
R_s	4.5	4.5	5.0	5.0	-	4.5	-	5.0

5th Overtone Resonators

Unit K-5-1

Time of Irradiation	0	5	10	15	30	162	292	1305
Frequency Shift (cycles)	Initial Value 19, 978, 766	-91	-133	-183	-208	-263	-286	+252
ppm	0	-4.6	-6.7	-9.2	-10.4	-13.1	-14.3	+12.6
R_s (Ohms)	5.0	5.5	6.0	6.0	-	6.0	5.5	5.5

TABLE I (Continued)

5th Overtone Resonators (Continued)

K-5-9-Ag 19, 980, 263		-61	-96	-115	-172	-232	-244	+247
Shift ppm	0	-3.1	-4.8	-5.8	-8.6	-11.6	-12.2	+12.3
R_s	5.0	5.5	5.5	5.5	5.0	5.0	5.5	5.5
K-5-10-Ag 19, 992, 261		-62	-98	-116	-160	-200	-219	+227
ppm	0	-3.1	-4.9	-5.8	-8.0	-10.0	-10.9	+11.4
R_s (ohms	5.0	5.5	5.0	-	5.0	5.0	5.0	-

Note: Resonators were exposed to Ce-137 radiation at 1.6 x 10^6 rad/hours. They were retrieved from the well and allowed to cool five minutes (to air conditioned room temperature for each measurement)

ARPA Order No. 54-60
Engineering Experiment Station
Georgia Institute of Technology
10 June 1960 to 30 June 1961

Contract No. DA-36-039-sc-85363
(Unclassified)

Stability Studies of Quartz Crystals for Satellites

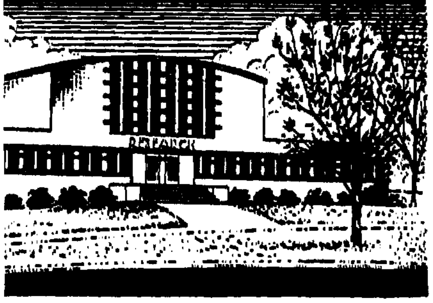
By

R. B. Belser and W. H. Hicklin

Report No. 1 (First Quarterly)
Contract No. DA-36-039-sc-85363
10 June 1960 to 1 October 1960
Georgia Tech Project No. A-508

Placed by the U. S. Army
Signal Research and Development Laboratories
Under ARPA Order No. 54-60

"The work performed under this contract was made possible by the support of the Advanced Research Projects Agency under Order No. 54-60 through the U. S. Army Research and Development Laboratory."



Engineering Experiment Station
Georgia Institute of Technology
Atlanta, Georgia

ARPA Order No. 54-60
Engineering Experiment Station, Georgia Institute of Technology
10 June 1960 to 30 June 1961

$52,489.00, Contract No. DA-36-039-sc-85363
(Unclassified)

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TABLE OF CONTENTS

LIST OF FIGURES

I. PURPOSE

The purpose of this project is to develop AT-Cut quartz resonators of 100 Mc frequency with stabilities suitable for operation in satellites or other space vehicles. Initial target stabilities are 0.3 ppm per year and ultimate desired stabilities are 0.1 ppm per year. These are to be maintained in spite of temperature cycling over the range 0° to 60°C one to several times per day and in spite of exposure to radiation typical of that of the Van Allen radiation belt.

Resonators operated in the 5th, 7th and 9th overtone modes will be investigated. Plating materials are to be silver or aluminum. Effects of radiation similar to that in the Van Allen belt on the stability of the 100 Mc resonators will be determined.

II. ABSTRACT

The purpose of this project is to develop 100 Mc resonators able to maintain frequencies which vary less than three parts in 10^7 per year with an ultimate objective of less than one part in 10^7 per year.

Ovens for storage of groups of 36 or less 100 Mc quartz resonators at 0°C, 60°C and for cycling them through the temperature range 0°C to 60°C one or more times per day have been completed. Instrumentation for measurement of these units, while stored at the specified temperature, to $\pm$ 2 parts in 10^8 has been developed. In addition, a calibration system for the 0-76-U frequency standard, based on the 18 KC signal received at Atlanta, Georgia, from radio station NBA, Balboa, Canal Zone, accurate to within 6 parts in 10^{10}, has been established.

Thirty-seven 100 Mc resonators have been fabricated. These consisted of 18 fifth overtone units plated with Al, 6 seventh overtone units plated with Al, 6 ninth overtone units plated with Al, and 7 ninth overtone units plated with silver. Because of experimental difficulties, many of the frequency data collected this quarter are unreliable. However, one unit, a ninth overtone unit base plated only with silver, was shown to have drifted less than nine parts in 10^8 in 45 days. Other similar units exhibited a similar stability. Drifts registered for most units were a few parts in 10^7 for the same period. The measurement techniques which are under continued development are expected to yield more consistent data during the next quarter.

III. PUBLICATIONS, LECTURES, REPORTS AND CONFERENCES

No publications, lectures or reports were made during the first quarter other than normal monthly letter reports.

Mr. Richard B. Belser, of the Georgia Institute of Technology, project director, visited the U. S. Army Signal Research and Development Laboratories at Fort Monmouth, N. J., for a conference on the project with Dr. G. K. Gutwein, Mr. M. Bernstein, Mr. J. Stanley and Mr. P. E. Mulvihill on 1 July 1960. The program and facilities available for the project were discussed. It was agreed that work would proceed essentially as outlined in the proposal. Mr. Belser presented a summary of aging data and explained the correlation between leak measurements obtained by the vacuum oil leak test and the stabilities obtained for 600 industrially procured 16.5 Mc resonators.

Subsequently on 31 August 1960 a second conference was held at the same place between the same parties with the exception of Mr. Stanley. Progress to date and procurement sources for better 100 Mc quartz blanks were discussed. Plans for the continuation of a parallel aging study of 16.0 Mc resonators at the fundamental frequency and at the third and fifth overtone modes were outlined.

IV. FACTUAL DATA

A. Introduction

The work on stable resonators for oscillator operation at or near 100 Mc/sec was anticipated some 16 months in advance of the award of this project. As a result, preliminary work on the building of necessary constant temperature ovens and a frequency measuring system of the necessary degree of accuracy and rapidity of measurement were developed in part under preceding Contract No. DA-36-039-sc-78905 and in part under funds provided by the Georgia Institute of Technology.

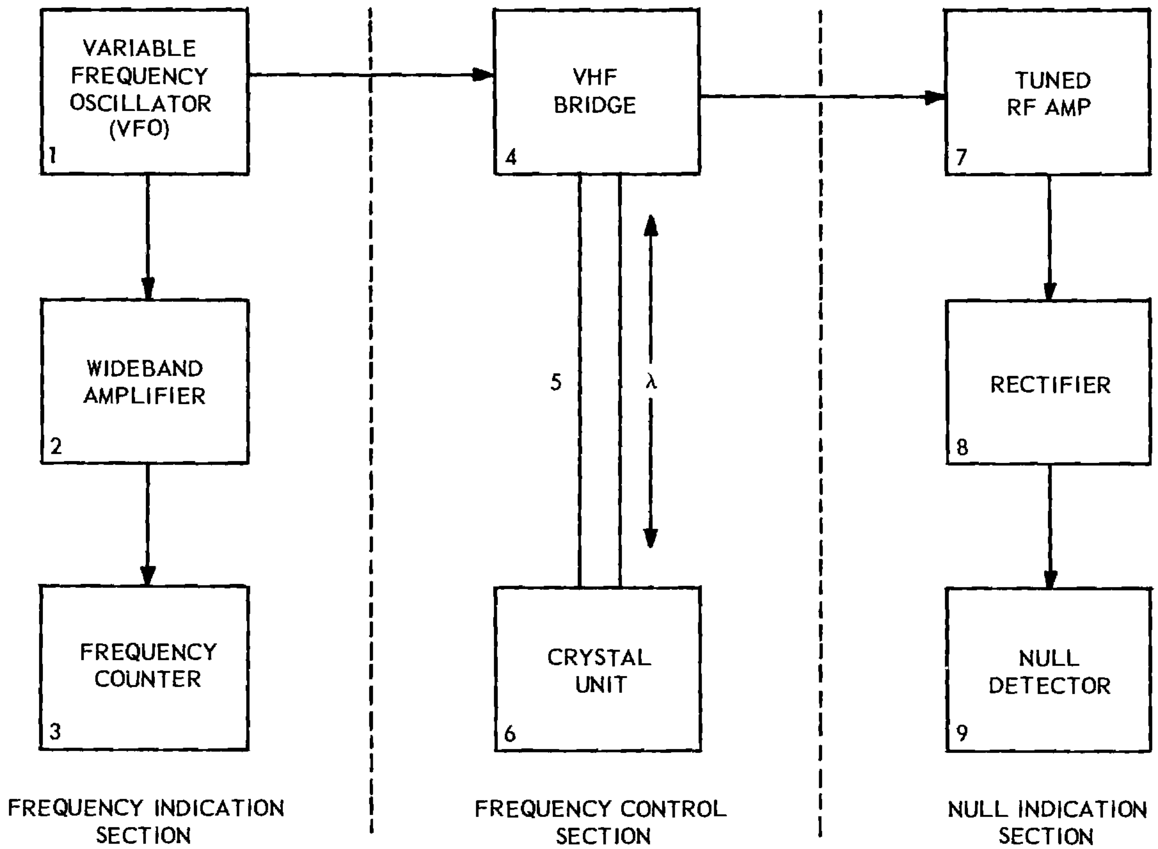
Progress on the development of this equipment has been reported in the Interim Report of the subject contract dated 30 June 1959, and in subsequent Quarterly Reports (especially No. 2 dated 30 September 1959). The Final Report of Contract No. DA-36-039-sc-78905 detailed the progress in instrumentation to 1 July 1960, the beginning of the present contract.

B. Apparatus

1. Apparatus on Hand

The apparatus already on hand, as outlined in the reports cited in the preceding paragraph, consisted essentially of an oscillator, the CI Meter TS-15 (or equivalent) for driving the crystal, a VHF bridge and associated instruments for adjusting the crystal to resonance by means of a null detector, an amplifier and a frequency counter. A block diagram of the system is shown in Figure 1. For connection to resonators in the ovens a tuned coaxial line was required.

Ovens for maintaining a constant temperature of 55°C and temperature cycling over the range 0° to 55°C had previously been constructed to hold 36



1. TS-15 (OR EQUIVALENT)
2. IFI MODEL 530
3. HEWLETT-PACKARD MODEL 524C
4. VHF BRIDGE (See Text)
5. FULL WAVE (100 MC) TWIN-COAXIAL LINE
6. CRYSTAL UNIT FOR TEST
7. VHF RECEIVER, SERVO MODEL R-5200
8. RECTIFIER (See Text)
9. HONEYWELL MODEL 104W1G

Figure 1. Block diagram for frequency measuring equipment for 100 Mc/sec.

resonators each, and only required installation of 60°C thermostats in order to fit them for the 60°C operating temperature level finally established for this work.

A complete technical description of the measuring equipment and ovens is contained in Quarterly Report No. 2 and also in the Final Report on Contract No. DA-36-039-sc-78905, dated June 30, 1960.

2. Oven for Resonator Storage at 0°C

In addition to the apparatus described above, an oven for operation at 0°C was constructed. This, like the other ovens previously constructed, consisted of five nested aluminum boxes with 1/4" felt interliners and contained positions for 36 resonators. The 0°C temperature was obtained by placing the oven in a freezer chest and setting the oven's thermostat to maintain the temperature at 0°C.

In order to make the oven accessible, the top of the freezer was removed and replaced with three-inch slabs of foamed plastic. The plastic was sectioned in such a manner that only a small portion need be removed in order to gain access to the oven.

3. Vacuum Fabrication Facilities

a. Base Plating Apparatus

The plating chamber was provided with a cold trap consisting of a monel tube of one inch I.D. to which copper fins were silver soldered. The trap, when filled with liquid nitrogen, is able to reduce the pressure in the chamber from 10^{-5} mm of mercury to approximately 10^{-6} mm of mercury.

Also within the chamber was placed a separate filament circuit from which a getter material could be evaporated. Titanium was found to have the best gettering action of all the materials tried. These included aluminum, zirconium, barium and misch metal. A series of baffles prevented the getter vapor from entering the section of the chamber where the crystal plating was done.

Pressures as low as 3×10^{-7} mm of mercury were obtained, using the combination of getter and cold surface pumping. It is anticipated that these and other improvements now under way, will reduce the minimum pressure in the plating chamber below 10^{-7} mm of mercury.

b. Final Plating Apparatus

A cold trap similar to the one for the base plating chamber and a getter filament was added to the final plating chamber. The copper fins of the cold trap served, in this case, as the baffle which prevented the getter vapor from entering the quartz plating section.

c. Vacuum Baking Apparatus

This apparatus was likewise equipped with a cold trap and getter filament arrangement within the vacuum chamber itself. Since a vacuum baking normally lasts several hours the capacity of the cold trap and the number of getter filaments were increased beyond that described for the plating apparatus. The trap was constructed from a copper tube three inches in diameter by two inches in height. Copper fins were silver soldered to the copper tank to increase the available cold surface and to serve also as the getter vapor baffle. The tank is filled by means of a monel tube of one-half inch I.D.

The getter filament was arranged in four sections so that evaporation could be made from one section at a time. This system without gettering has been evacuated to 4×10^{-7} mm of mercury.

The crystal units are to be coupled into the chamber by means of neoprene sealed packing glands. These are currently on order.

All of the vacuum chambers used for crystal unit fabrication have been provided with quick coupling devices so that a hot filament ion gauge tube may be inserted directly into the chamber to measure pressure.

d. Vapor Degreaser

An excellent, open top, vapor degreaser has been constructed as outlined below.

A two-turn coil of one-fourth inch O.D. copper tubing was soldered to the uppermost portion of a one-gallon, stainless steel container. About one pint of solvent placed in the container serves as the degreasing agent. Cold water is cycled through the copper coil and the solvent heated to the proper temperature. The cool zone formed near the top of the container traps most of the vapor so that the degreaser may be used without a top or elaborate venting. Trichlorethylene has been used as the solvent to clean filament assemblies, glassware, tools and related accessories with excellent success.

4. Standards for Accurate Frequency Measurement*

a. Introduction

Long term observations of the three O-76-U oscillators at Georgia Tech indicate that frequency deviations as large as 1 part in 10^8 may take place in one day's time. This points out the need for some external frequency references with which to calibrate the frequency of these oscillators.

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* This section contributed by W. B. Warren, Jr., Special Research Engineer, Georgia Institute of Technology.

Recent tests reported in literature[1] indicate that excellent calibration of local frequency standards may now be obtained by direct comparison of these local standards against the signals received from any one of several very-low-frequency standard signals. This method was selected as one which would be investigated as to its feasibility in this location.

There are currently available three such standard frequency broadcasts in the United States. Two of these are supplied by the National Bureau of Standards at Boulder, Colorado, on 20 KC and 60 KC. The other standard signal is provided by the Naval Research Laboratory through Radio Station NBA on 18 KC transmitted from Balboa, Panama Canal Zone. The frequency of each of these signals is maintained to within 2×10^{-10} of absolute frequency at the source.

In the case of the 18 and 20 KC signals, the basic source is a crystal oscillator which is continuously monitored against atomic frequency standards, while the 60 KC signal is derived directly from an atomic frequency standard. Due to the exceptionally low attenuation at these low frequencies, reception is by ground wave propagation and the attendant phase stability is excellent. Of these three signals, the 18 KC signal from Balboa is, by far, the strongest in Atlanta, Georgia.

b. Apparatus

Equipment has been constructed to permit a calibration of the O-76-U oscillators currently in use as the standard of frequency at Georgia Tech. Essentially this equipment consists of a TRF receiver, tuned to 18 KC,

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1. Pierce, John A., "Intercontinenta Frequency Comparison by Very Low Frequency Radio Transmission", Proc. IRE, pp 794-803, June 1957.

whose output is used to drive one X/input of a phase detector; the reference input is an 18 KC signal derived from the local O-76-U oscillator. The output of this phase detector is supplied to a low pass filter whose cut-off frequency is approximately 0.05 CPS; this reduces the overall noise band width for the system to 0.1 CPS. This extremely narrow band width insures clean recovery of the phase difference information.

This phase difference information is used to drive a strip recorder to provide a continuous record of the phase difference between the local standard and the received 18 KC signal from Balboa. Inspection of this record then permits an accurate determination of the absolute frequency of the O-76-U oscillator at any time.

The equipment arrangement for making frequency comparisons is outlined in more detail in Figure 2. A ferrite loop antenna is used to pick up the low frequency signal. Such an antenna has the advantage of small size, and its inherent band pass response aids in the suppression of high level adjacent channel interference. In addition, the ease of impedance matching which may be obtained with this antenna permits the placement of the antenna at a favorable position, which may be remote from the actual receiver.

The receiver proper consists of a main amplifier which contributes a gain of approximately 300,000 and an output amplifier of gain 100, which is used to drive the phase detector. The reference signal for the phase detector is derived from the 100 KC standard frequency input by dividing by 100 to get 1 KC, and multiplying this by 18 to obtain the designed 18 KC output.

This process is illustrated in the block diagram of Figure 2. The output of the phase detector is fed to a DC amplifier which raises the signal to a sufficient level to drive the strip recorder. The entire equipment has been

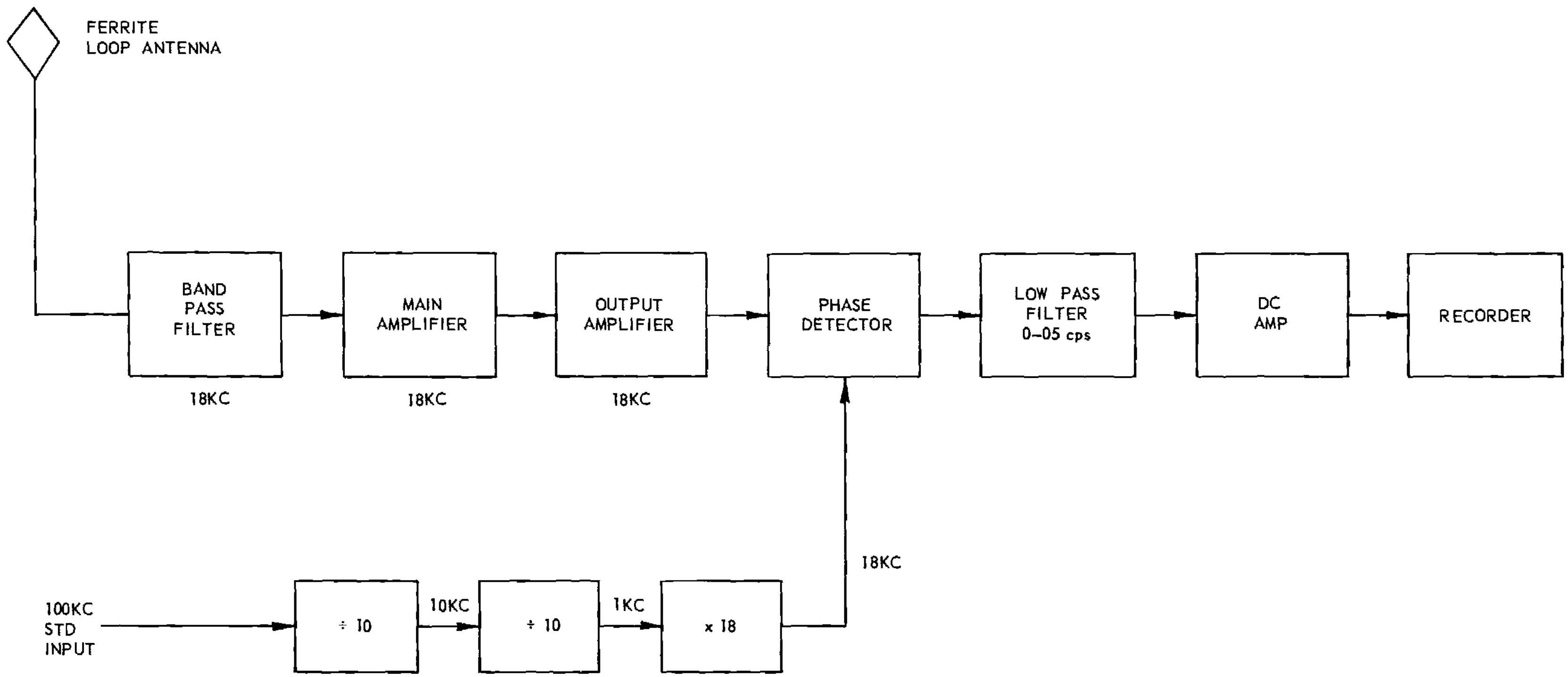

Figure 2. Equipment arrangement for making phase comparison at 18 KC.

rack-mounted and placed immediately adjacent to the 0-76-U oscillators to permit rapid calibration of these oscillators.

In an effort to obtain some measure of the degree of accuracy with which such calibrations of a local frequency standard could be made, the local phase reference was supplied by an Atomichron instead of the 0-76-U oscillator. When this was done, a record showing a frequency difference of approximately 80 parts in 10^{10} was obtained. Since the transmitted frequency of the 18 KC signal is maintained 150 parts in 10^{10} low with respect to Ephemeris Time* and since the frequency synthesizer by the Atomichron oscillator is 76 parts in 10^{10} low in respect to Ephemeris Time, the beat frequency should have been 74 parts in 10^{10}. Thus, the actual beat period obtained is in error by only 6 parts in 10^{10}.

The graph of Figure 3 shows the total number of beat cycles obtained as a function of time for a period of approximately 80 hours. Any deviation of the data points from a straight line indicates long term phase instabilities. From the figure, it is clear that this system of comparison can be relied upon to an accuracy of at least 6 parts in 10^{10}. This represents a considerable improvem is site heretofore.

C. Experimental Work

During the quarter thirt .cated. These consisted on 30 units t units base

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* Ephemeris Time is time bas h about the sun.

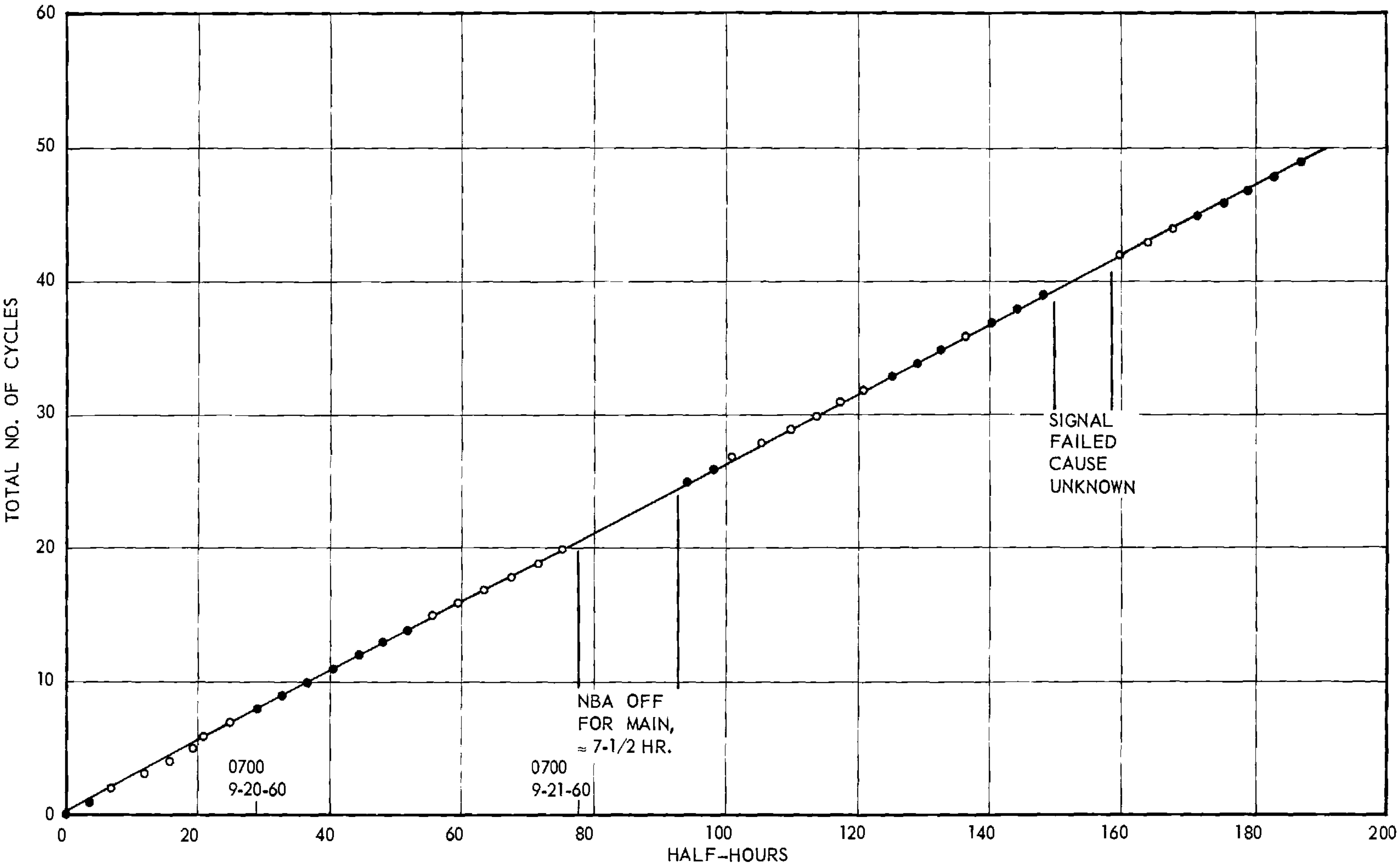

Figure 3. Data indicating number of beat cycles obtained by comparison of received 18 KC signal to atomichron signal over a period of 80 hours. The 18 KC signal originated from Radio Station NBA, Balboa, Panama Canal Zone.

plated only with silver. These were divided under the various overtone designations as follows:

5th overtone	18 plated with Al
7th overtone	6 plated with Al
9th overtone	6 plated with Al and 7 with Ag

All of these units were fabricated in glass containers after the fashion previously described for Al and Ag units respectively under Contract No. DA-36-039-sc-78905. None were overplated to frequency initially; thus overplating was eliminated as a variable for the first measurements.

Measurements were conducted with great care, but a number of variables such as temperature variations of the coaxial connector cable, connector contact pressure and lack of impedance matching between the connector cable and the units resulted in an overall apparent measurement accuracy little better than one part in 10^7.

The units of high resistance gave relatively good stability. The pattern of a typical unit is observed in Figure 4. Units of this type were more consistent in behavior and measurements appeared to be accurate to within about five parts in 10^8. However, this unit has drifted less than one part in 10^7 in 45 days.

Drifts for a better unit of each group except one are shown in Figures 5, 6, 7, 8, and 9. In the missing group no consistent measurements for a single resonator were obtained. The units in this group were of low resistance (about 25 ohms) and the poor quality of the measurements is attributed primarily to lack of impedance matching with the coaxial connector line.

The drift of unit B-9-2 of Figure 4 represents one of the better units fabricated. It is to be noted that this unit is a ninth overtone unit coated

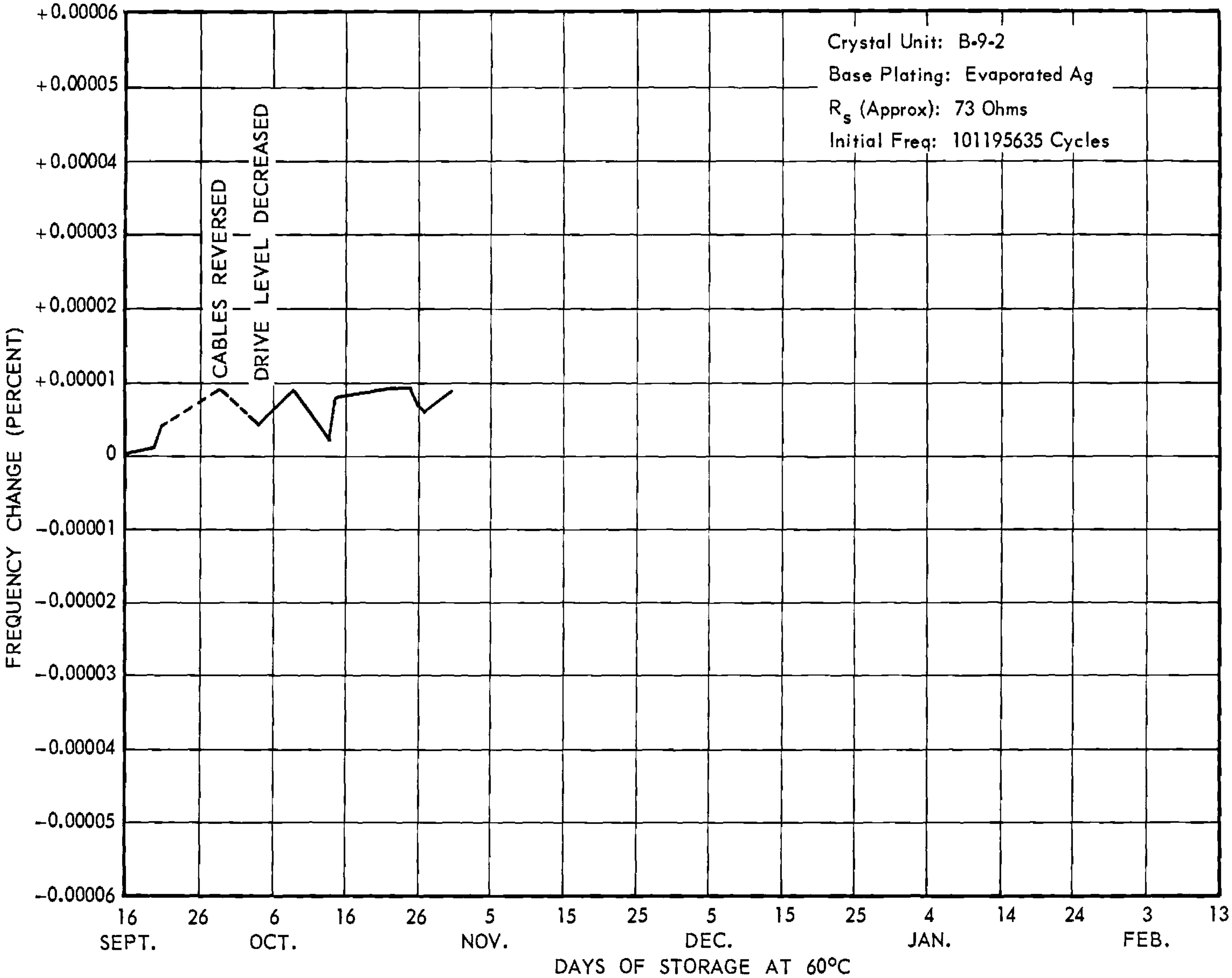

Figure 4. Frequency versus time data for resonator B-9-2, a ninth overtone unit coated with silver.

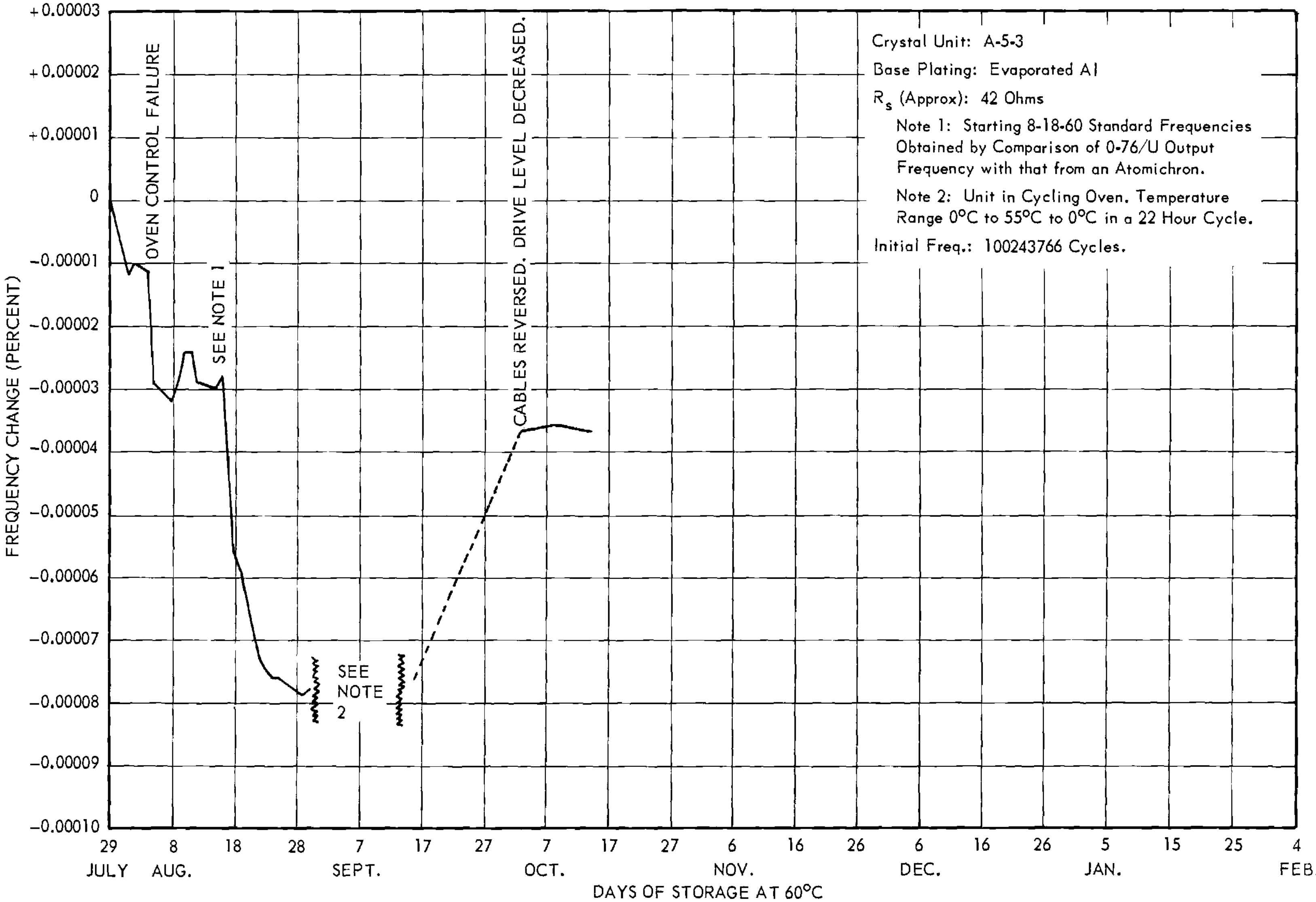

Figure 5. Frequency versus time data for resonator A-5-3, a fifth overtone unit coated with Al.

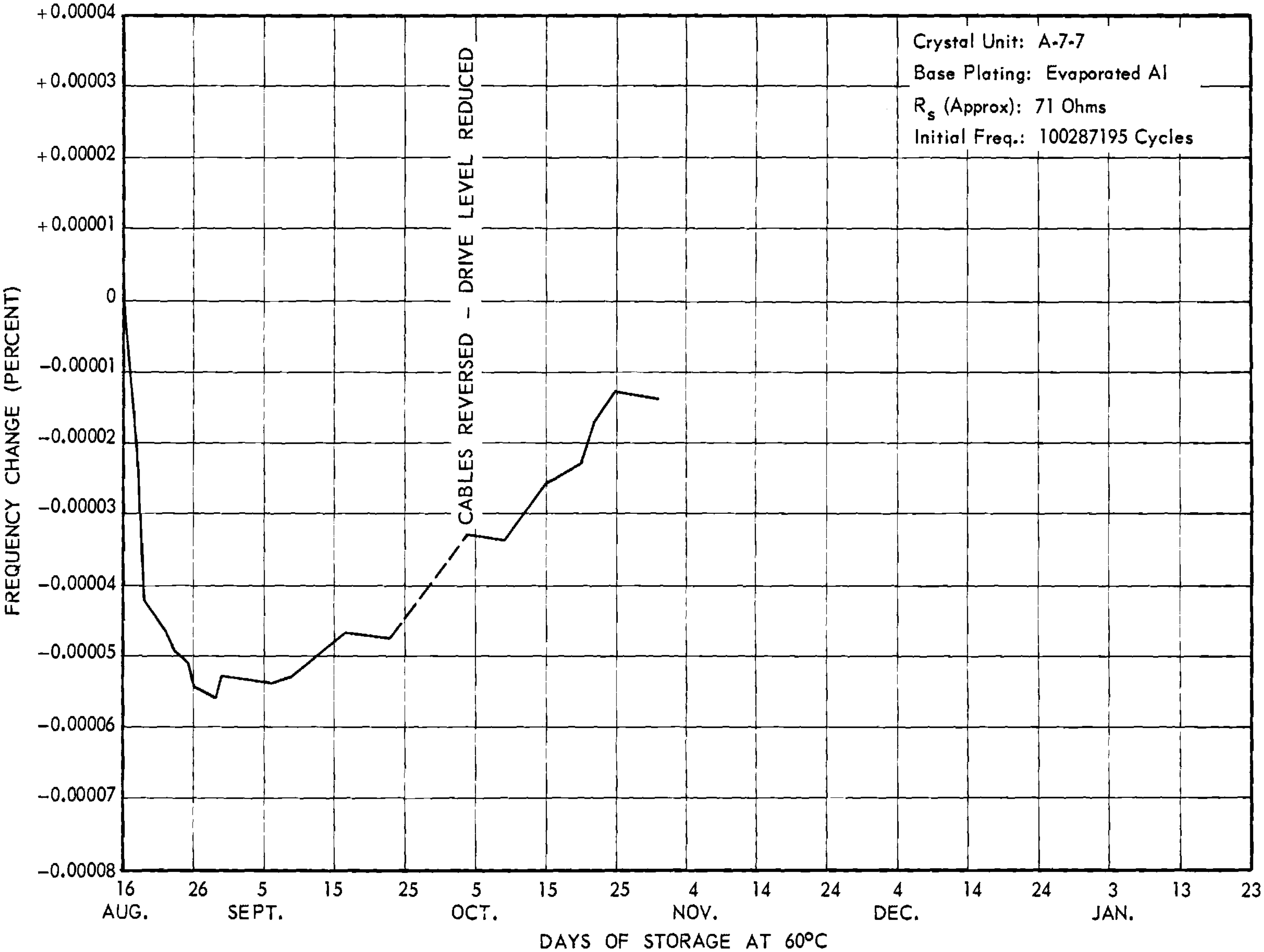

Figure 6. Frequency versus time data for resonator A-7-7, a seventh overtone unit coated with Al.

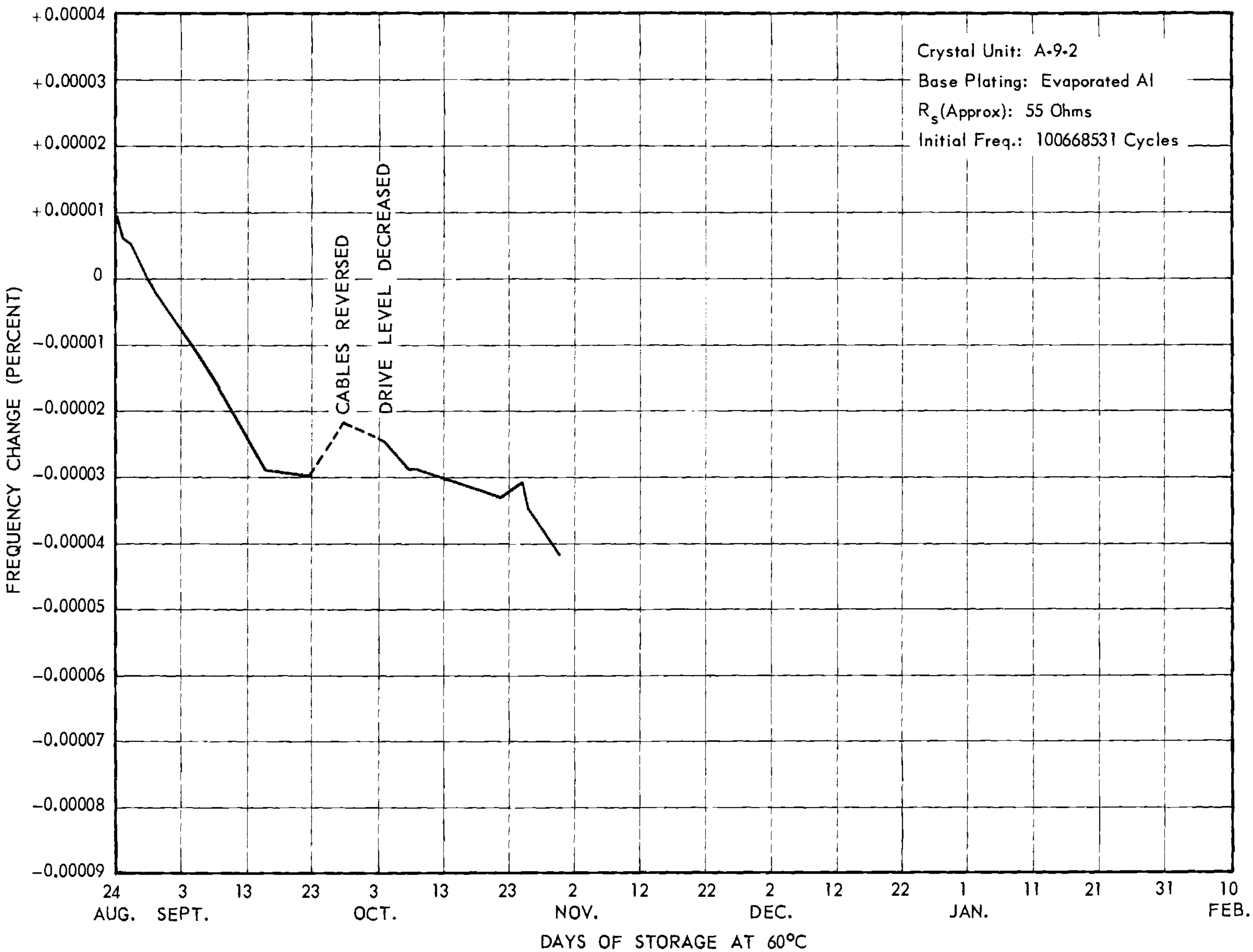

Figure 7. Frequency versus time data for resonator A-9-2, a ninth overtone unit coated with Al.

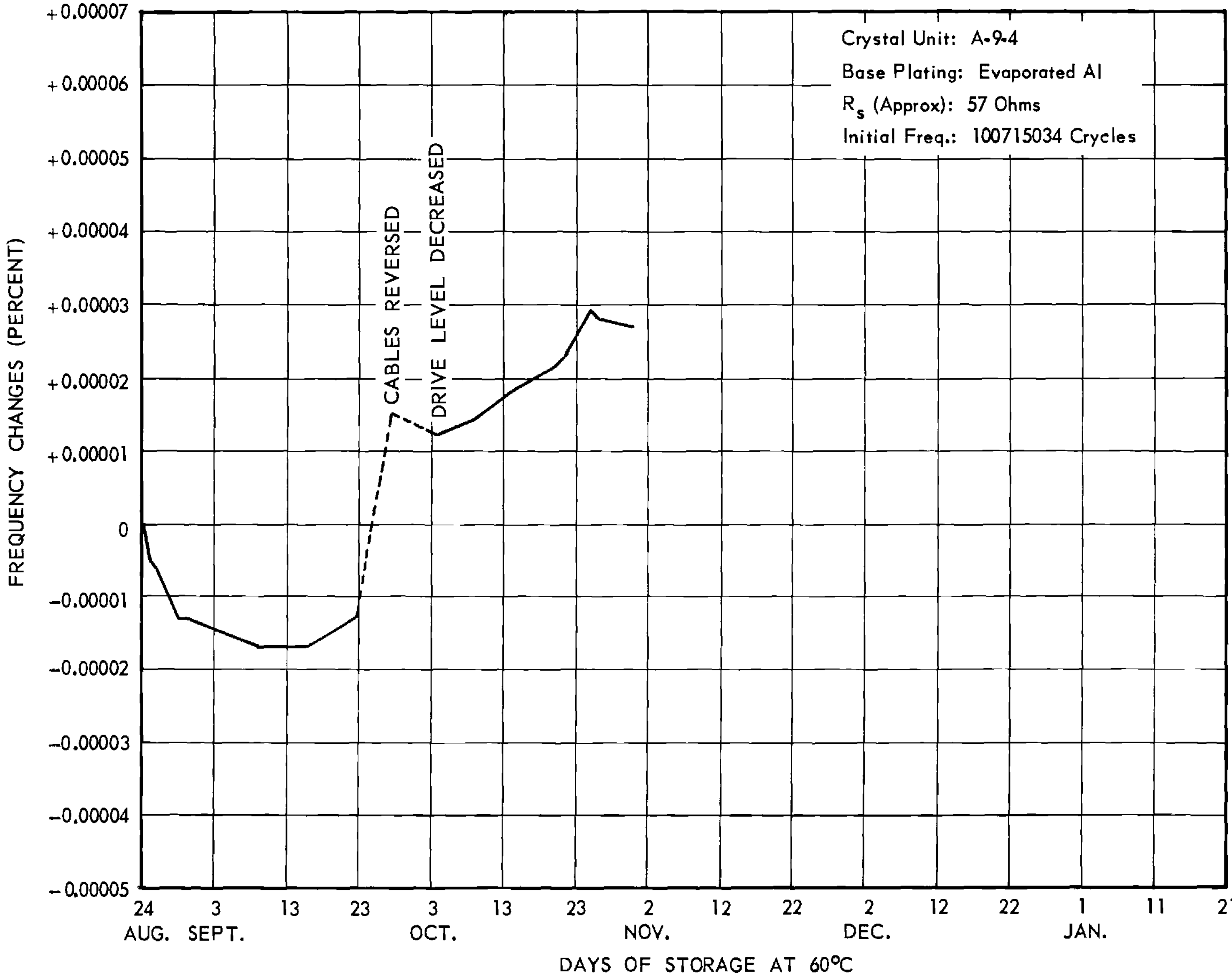

Figure 8. Frequency versus time data for resonator A-9-4, a ninth overtone unit coated with Al.

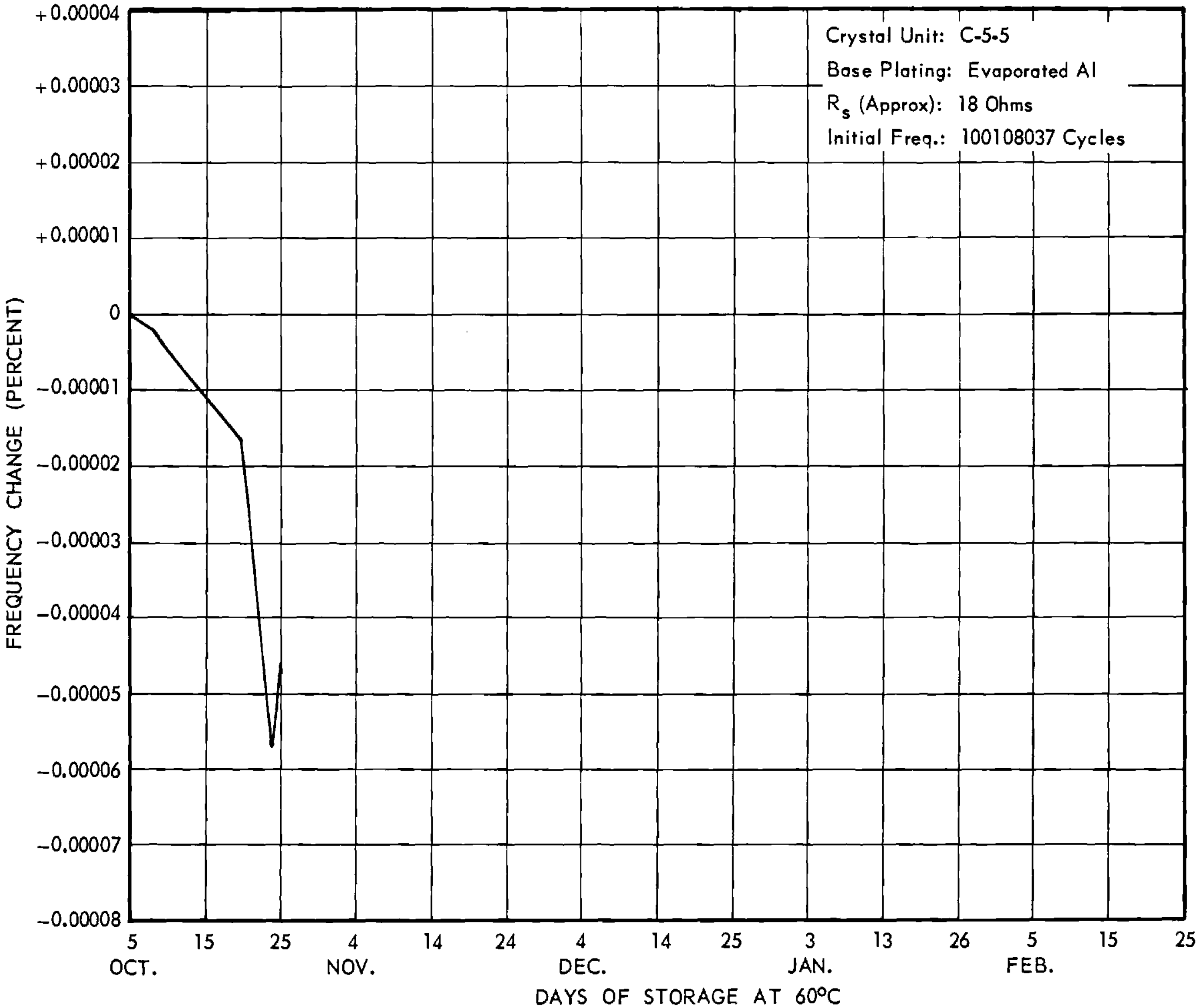

Figure 9. Frequency versus time data for resonator C-5-5, a fifth overtone unit coated with Al.

with silver. Very recent measurements on similar units made with greatest care as to time, room temperature, and procedures have shown measurements repeatable to one or two parts in 10^8. Most of the units examined, however, have not performed so stably and are typified in the figures previously mentioned. There is reason to believe, however, that better measurements will be forthcoming now that these initial studies have been made.

Figures 8 and 9 show the performance of two ninth overtone units coated with high purity (99.999%) aluminum. The drift direction of unit A-9-2 is down and that of A-9-4 is up. In fact, after six weeks the latter unit registers a total drift of only + 2.7 parts in 10^7. Unit A-9-4 seems to be reasonably stable since the frequency drift appears to be leveling off presently.

D. Comments

A large part of the current quarter has been devoted to perfection of apparatus and measurement techniques. Although some 37 resonators were fabricated and tested, many of the tests were run before satisfactory measurement techniques had been worked out. As a result the data show a number of abrupt shifts not normal to proper aging data. Some of the experimental difficulties which have been encountered are described below.

The VHF bridge frequency measuring system did not prove initially to be more precise than $\pm$ 1 part in 10^7. The trouble has been traced in part to the sensitivity of the resonant line to changes in room temperature. Two courses of action are being considered: (1) to control the room temperature to within about $\pm$ 1°C; (2) to provide compensation to readjust the line to resonance when necessary.

Another problem appears to be impedance matching between the coaxial line and the resonator itself. The coaxial line as now connected has approximately 100 ohms impedance. The most consistent data has been obtained from resonators of high resistance.

With adequate control of temperature, impedance matching, and level of the driving signal, the equipment is believed capable of making measurements repeatably to about $\pm$ 2 parts in 10^8.

The calibration of the O-76-U frequency standards based on the NBA signal of 18 KC received from Balboa, Canal Zone, has proved quite feasible. However, the availability of the Atomichron* as an additional frequency source has proven of great value in verifying the stability of the received signal and as a standard of absolute frequency. Considering the accuracy of measurements required or anticipated it is believed that an Atomichron should be made available to the project on a continuous basis.

- - - - -

* The Atomichron, now here, is available only on a short term loan basis.

V. CONCLUSIONS

The strength of the 18 KC signal from radio station NBA, Balboa, Canal Zone, is received with sufficient strength in Atlanta, Georgia, to provide an accurate signal for calibration of a secondary frequency standard such as the Western Electric O-76-U. Accuracy of comparison better than six parts in 10^{10} has been demonstrated.

Measurement accuracy and repeatability for the measurement system developed for measurement of resonators stored in various ovens were not sufficient to obtain useful aging data during the current quarter. Variation in room temperature, impedance matching between the coaxial connector line and resonator, drive level, and in methods of making contact reduced accuracy to little better than $\pm$ 1 part in 10^7. With careful attention to each of the details measurements to within $\pm$ 2 parts in 10^8 appear feasible.

Of the resonators measured ninth overtone units coated with silver gave the highest stability, a positive drift of approximately 9 parts in 10^8 in a 45-day period.

VI. PROGRAM FOR THE NEXT INTERVAL

Frequency measurement conditions and techniques will be improved to maintain an accuracy of $\pm$ 2 parts in 10^8.

Resonators of the 5th, 7th and 9th overtones will be base plated with silver or aluminum and overcoated to frequency with the respective metal. Storage and measurement of resonators at 0°C and cycling the units over the temperature range 0°C to 60°C will be commenced. Plans are proceeding for the exposure of suitable units to radiation as soon as a proper series of aging measurements for units in oven storage have been completed.

VII. IDENTIFICATION OF KEY TECHNICAL PERSONNEL

The following persons have been employed on this project during its first quarter for the times indicated.

Name	Position	Time (Hours)
Richard B. Belser	Project Director	177
Douglas W. Robertson	Research Engineer	66
W. Bruce Warren	Research Engineer	187
Samuel N. Witt	Research Engineer	18
Walter H. Hicklin	Ass't. Research Engineer	541
James O. Darnell	Research Assistant	445
Walter L. Reagh	Research Assistant	33
W. Donald Dawson	Student Assistant	480

Mr. Belser has been associated with resonator aging studies sponsored by USASRADL for over ten years and has been assisted by Mr. Hicklin for approximately nine years. Mr. Robertson, Mr. Warren and Mr. Witt, electrical engineering graduates of Georgia Tech with M.S. degrees, have served in the capacity of project directors on projects sponsored by both Signal Corps and Air Force Agencies for a number of years and each has had recent industrial experience with leading communication manufacturers. The primary responsibility of Mr. Warren and Mr. Witt has been to establish the frequency standard calibration arrangement described in this report.

Respectfully submitted,

Richard B. Belser
Project Director

Approved:

Vernon Crawford
Head, Physics Branch
Physical Sciences Division

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Georgia Institute of Technology
10 June 1960 to 30 June 1961

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(Unclassified)

Stability Studies of Quartz Crystals for Satellites

By

R. B. Belser and W. H. Hicklin

Report No. 2 (Second Quarterly)
Contract No. DA-36-039-sc-85363
1 October 1960 to 1 January 1961
Georgia Tech Project No. A-508

Placed by the U. S. Army
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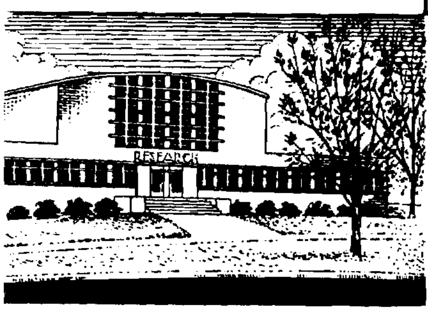

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Georgia Institute of Technology
10 June 1960 to 30 June 1961

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I. PURPOSE

The purpose of this project is to develop AT-Cut quartz resonators of 100 Mc frequency with stabilities suitable for operation in satellites or other space vehicles. Initial target stabilities are a maximum frequency deviation of $\pm$ 0.3 parts per million per year and ultimate desired stabilities of $\pm$ 0.1 parts per million per year. These stabilities are to be maintained while the resonator is subjected to temperature cycles of 0° to 60°C one or more times per day and exposed to radiation similar to that of the Van Allen belt.

Resonators operated in the 5th, 7th and 9th overtone modes will be investigated. Plating materials are to be silver or aluminum. Effects of radiation similar to that in the Van Allen belt on the stability of the 100 Mc resonators will be studied.

II. ABSTRACT

The purpose of this project is to develop 100 Mc resonators able to maintain frequencies which vary no more than $\pm$ 0.3 parts per million per year with an ultimate objective of no more than $\pm$ 0.1 parts per million per year.

Primary effort during the quarter was directed toward improvement of the frequency measuring technique to assure a precision near $\pm$ 1 part in 10^8 for routine frequency measurements. Improvements of the previously used VHF bridge by temperature control of the bridge itself, better coaxial lines and connectors, and reductions of voltage irregularities supplied to the screen grid of the oscillator tube of the CI Meter TS-15, brought the precision of the measurements approximately to the desired value. Simultaneously a second capacitance bridge, isolated from the oscillator by a ferrite transformer and having one terminal of the crystal grounded, was designed and constructed. This unit, in initial tests has given comparable accuracy. It has the additional feature of allowing voltage drops across the crystal to be directly monitored and thus allows accurate drive level settings.

Seventy crystal units have been fabricated. These consisted of 10 5th overtone units base plated with aluminum and final plated with silver, 10 with silver only and 20 with copper only. 30 9th overtone units plated with silver only were also fabricated. Aging measurements have been continued for units stored at 60°C. At least three 9th overtone units plated with silver have maintained the target stability of 1 part in 10^7 in 120 days and show no definite directional drift. The new measurement techniques should now allow a determination of the true stabilities of these units.

III. PUBLICATIONS, LECTURES, REPORTS AND CONFERENCES

No publications or lectures were made during the second quarter. Monthly letter reports and Report No. 1 (First Quarterly) were completed and submitted. A summary of a proposed paper for the fifteenth annual Frequency Control Symposium to be held at Atlantic City, N. J. 31 May 1961 was submitted for approval to the Director of the Frequency Control Division of the U. S. Army Signal Research and Development Laboratories.

Conferences held during the second quarter were by telephone only. Mr. R. B. Belser talked with Dr. G. K. Guttwein, Mr. P. E. Mulvihill and Mr. M. Bernstein during the course of the quarter. Measurement problems and the course of action to be taken were discussed.

IV. FACTUAL DATA

A. Introduction

The principal pieces of apparatus for fabrication, storage and measurement of the 100 Mc resonators under study were described in Report No. 1 (First Quarterly) of the Project and the Final Report of Contract No. DA-36-039-sc-78905 recently completed, 30 June 1960.

During the first quarter the inadequacies of the frequency measurement system for accuracies of the order of magnitude (a part in 10^8) required became apparent. In spite of ability to readily measure one part in 10^7 by the bridge method previously used, the extension of this method by an order of magnitude proved to be a difficult problem. A large part of this Quarter has been spent on measurement improvement. Fabrication and routine measurement activities have been continued whenever equipment modification did not interfere. The problem of measurement and steps taken to improve it are discussed in succeeding paragraphs.

B. Apparatus

The apparatus assembled during the Quarter has been constructed primarily in conjunction with the experimental work on the measurement problem. It will therefore be described in conjunction with the experimental work rather than in a separate section.

C. Experimental Work

1. The Measurement Problem*

In the measurement system currently in use, the crystal is remotely located from the bridge with which the measurement is being made. As a result the impedance presented to the bridge by the crystal is a function of the length of the connecting cables. Although the nominal length of these cables is fixed, small fluctuations in room temperature are sufficient to produce significant changes in cable length. This effect limits the accuracy with which the frequency of the crystal can be determined. In order to obtain some idea of the magnitude of the effect which must be measured, a

*This section contributed by W. B. Warren, Jr., Research Engineer, Georgia Institute of Technology calculation can be made of the phase angle change associated with a crystal frequency change of one part in 10^8, i.e., one cycle at 100 Mc.

In the immediate vicinity of series resonance, the net reactance of a crystal is given by the expression

$$X = R(2Q_o \frac{\Delta f}{f_o})$$

Where R = Series resistance of crystal

f_o = Series resonant frequency

Δf = Difference between exciting frequency and f_o

Q_o = Q evaluated at f_o

and the phase angle of the impedance is

$$\theta = \tan^{-1} (\frac{X}{R})$$

or

$$\theta = \tan^{-1} (2Q_o \frac{\Delta f}{f_o})$$

for small values of θ,

$$\theta = \tan^{-1} \theta,$$

or

$$\theta = 2Q_o \frac{\Delta f}{f_o}.$$

For the case under consideration:

if: Δf = 1 cycle

f_o = 100 Mcs.

Q_o = 10,000

then:

$$\theta = (2)\ (10^4)\ (10^{-8}) = 2 \times 10^{-4} \text{ radians.}$$

This is a very small change in angle, but it is, nevertheless, a much larger percentage change than is associated with the magnitude of the crystal impedance. Consequently, a system is required which will measure changes in the crystal phase angle of approximately 2×10^{-4} radians if the required accuracy is to be obtained. Fortunately, a measurement of this angle is within the capability of a well designed bridge technique. This small angle demonstrates why the previously mentioned small fluctuations in cable length, due to temperature, produce relatively large fluctuations in the measured data when these data are displayed on a scale sufficiently large to demonstrate changes of the order of one part in 10^8.

2. A Capacitance Bridge

To overcome the effect of cable length variation, another bridge has been constructed for use directly at the oven terminals. Although these terminals are several inches removed from the actual crystal, the connecting cables between these terminals and the crystal are inside the oven and do not experience temperature fluctuations of any appreciable magnitude. The form of this bridge is indicated in Figure 1.

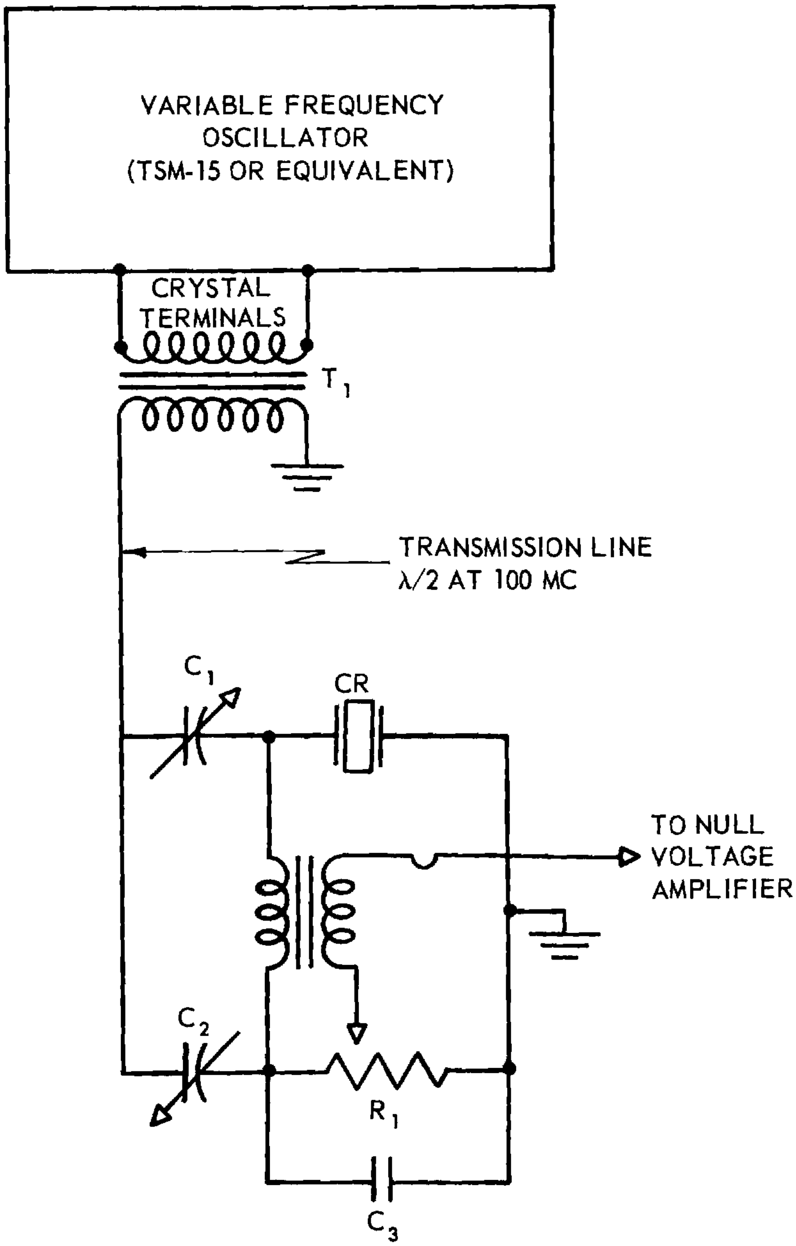
The bridge is a conventional capacitance bridge, the capacitance form being chosen because of the difficulty of obtaining satisfactory high frequency variable resistors. Instead of the conventional balanced-to-ground arrangement required for use with most CI meters, a single ended arrangement is employed; this eliminates the need for a balanced coaxial system to drive the bridge. In addition, this arrangement permits the operation of the crystal with one grounded terminal thus minimizing the effect of stray capacitance and simplifying the measurement of crystal drive level. The unbalanced arrangement is made possible by the use of a balanced-to-unbalanced transformer which is driven directly from the crystal terminals of the CI meter. This transformer has a ferrite core and unity turns ratio to permit almost unity coupling to be obtained, and therefore reflects impedances in the secondary to the primary essentially unchanged.

This unbalanced system has been constructed using the ferrite core transformer and has been used to give crystal control of the CI meter with the bridge operated at the end of a cable several feet in length. Preliminary tests indicate that the sensitivity of the system is sufficient to detect the desired changes in crystal frequency, i.e., one part in 10^8*.

3. Other Approaches to the Measurement Problem

A conference between the members of the project staff and associated special research engineers assigned to electronic research, revealed four

* Experiments made subsequent to the end of the 2nd Quarter have indicated that a precision of $\pm$ 1 part in 10^8 is obtained with this arrangement.



T_1 – FERRITE TRANSFORMER (UNITY TURNS RATIO)

C_1, C_2 – DIFFERENTIAL CAPACITOR (3–20 $\mu\mu f$)

R_1 – RESISTOR (100Ω)

CR – CRYSTAL RESONATOR

C_3 – COMPENSATING CAPACITOR (ABOUT 10 $\mu\mu f$)

Figure 1. Capacitance bridge for improved measurement of high frequencies.

possible experimental approaches to the solution of the accurate, rapid measurement of frequencies in the 100 Mc range to a part in 10^8.

a. Bridge with Four Coaxial Lines

Construct a VHF bridge similar to the one now being used, see Figure 2, but having four coaxial lines rather than two. One pair of lines will be used to connect the crystal to bridge, the other pair to connect the resistor (R_1) to the bridge. The lines are to be similar in all respects and to be one-half wavelength long at 100 Mc. With the resistor (R_1) connected to the bridge with a pair of coaxial lines, as is the crystal, the changes due to ambient temperature variation in the crystal loop will, in theory, be compensated by equivalent changes in the resistor loop.

b. Bridge at Oven Site

Connect the bridge to the CI meter with a pair of coaxial lines of one-half wavelength at 100 Mc. The bridge will then connect directly to the BNC terminals at the bottom of the crystal ovens.

The coaxial lines operated in this way should not cause frequency changes due to variation of the ambient temperature. The short coaxial leads of about five inches through the oven into the crystal sites should have quite constant properties being securely connected and held at a constant temperature.

c. Coaxial Lines with Air Dielectric

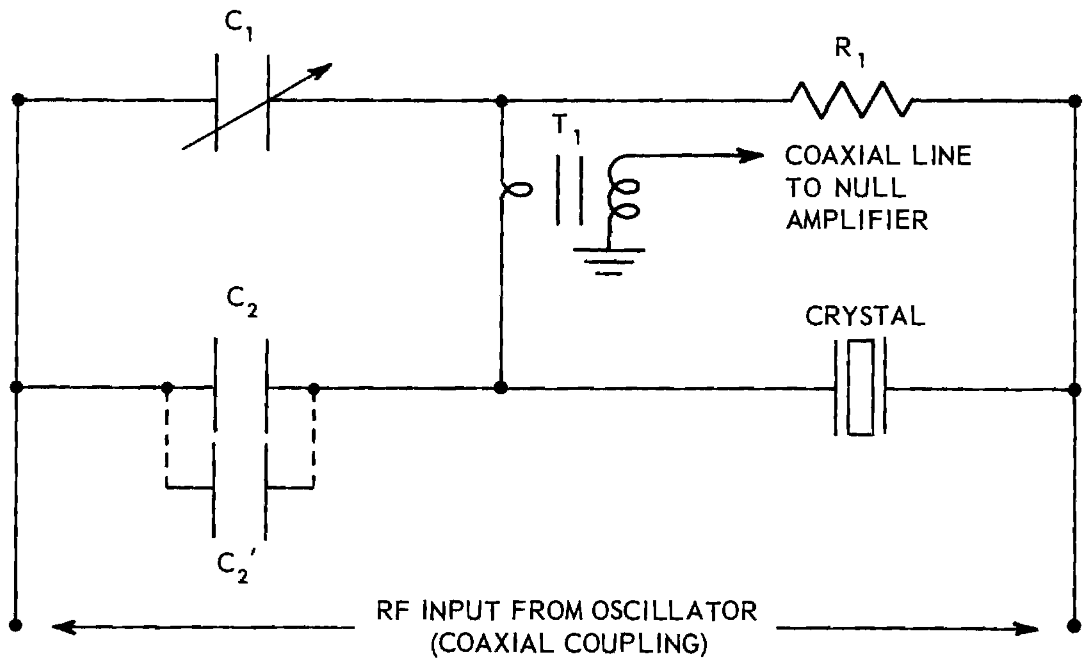
For the coaxial line* now used to engage the crystals, substitute a cable having air dielectric. Such cable, although presenting some problems due to lack of flexibility, should be less sensitive to ambient temperature change and would of course present less attenuation.

d. Passive Measurement System

A solution that involves equipment not presently available to the project was suggested. The method is similar to the "Transmission method**" of measuring the constants of piezo-electric vibrators reported

* Belden 8259 low noise cable.

** Engineering Report No. E-1116 by E. A. Gerber, U. S. Army Signal Research and Development Laboratories, Ft. Monmouth, N. J., 16 April 1953



C_1 – AIR DIELECTRIC 2.7 TO 19.6 $\mu\mu f$
C_2 – SILVERED MICA 10 $\mu\mu f$
C_2' – SILVERED MICA 15 OR 40 $\mu\mu f$ (See Text)
R_1 – COMPOSITION 120 Ω
T_1 – ONE TURN PRIMARY, TWO TURN SECONDARY, FERRITE CORE
CRYSTAL – PROVISION MADE FOR OPERATION DIRECTLY INTO BRIDGE OR AT END OF FULL WAVE TWIN-COAXIAL LINE

Figure 2. VHF Bridge for frequency measurement.

previously by Dr. E. A. Gerber of USASRDL. This method consists essentially of a passive one in which the crystal is scanned from an external frequency source over a range of frequencies near and including its resonant one. Meauurements of the impedance of the crystal allows one to plot a curve showing the frequency at which the impedance of the resonator becomes a minimum. This is the resonant frequency of the unit.

The detection system should be phase sensitive for greatest accuracy. A high-quality frequency synthesizer is required as the signal generator.

4. Experimental Examination of the Measuring Methods

The experiments listed under paragraphs a and b above were performed as noted below.

a. The Four Terminal Bridge

A four terminal bridge was constructed as suggested in Paragraph 3a above, i.e., a resistor and the crystal were connected to the bridge with half-wave coaxial lines of as near equal length as could be cut by hand. The bridge did not balance. A balance may have been attained by varying the length of the line in the resistor loop. This action was not taken, however, since it departed from the original intention of coaxial lines of equal length for each loop.

b. Reduction of Length of Coaxial Line to the Resonator

The suggestion that the bridge could be mounted immediately beneath the storage oven was examined by mounting the resonator at the bridge on short coaxial lines equivalent to the oven resonator leads (five inch lengths). Crystal control of the oscillator circuit was obtained but oscillation of the circuit also occurred without the crystal. This behavior indicated a low value of shunt reactance across the resonator.

5. Other Measurement Studies

In addition to the above experiments the following improvements or experiments were performed.

a. A New Half-Wave Line

A new half-wave line was made for use with the present measuring system. The same type coaxial cable was used*. However, BNC terminations UG-88C/U rather than UG-88/U were used. Some mechanical improvement was obtained.

b. Adjustable Line Stretchers

Adjustable line stretchers** were added in each coaxial line used to engage the crystal positions. The total line length was then adjusted to a full-wave at 100 Mc. Two methods were tried in an effort to obtain the necessary precision of adjustment. (1) The crystal was plugged directly into the bridge and balanced. The open circuited lines were then connected to the bridge and adjusted to length until the null was again obtained. (2) The crystal was measured (frequency and resistance) directly in the bridge; the crystal was then placed in a simulated oven position and the lines readjusted until the same frequency and resistance were obtained.

It was found that the frequency of a given crystal could be measured no better than about $\pm$ 5 pp 10^7 on repeated line calibration adjustments. As a result, the system was temporarily discarded.

The line stretcher assembly was designed so that each stretcher was adjusted separately. It now appears likely that the stretchers should adjust together.

c. Constant Screen Voltage Supplied CI Meter Tube V-4

Although the frequency measuring equipment obtained power through a constant voltage transformer*, the crystal drive level was slightly

* Belden No. 8259.

** General Radio No. 874-LK20 constant impedance adjustable lines.

*** Sola, Cat. No. 20-13-150, Type CV-1.

sensitive to line voltage variations. To reduce such effects, the screen of tube V-4 of the CI meter TSM-15 (see Handbook of Instructions for the Crystal Impedance Meter TSM-15) was supplied voltage from a 45V battery and an associated voltage divider circuit. The voltage divider circuit was connected across the battery and the screen voltage was taken from across a 5 K ohm helipot.

d. Constant Temperature Oven for Bridge

It has been demonstrated in recent weeks that the VHF bridge itself is more sensitive to ambient temperature changes than had been expected. To reduce such effects a simple oven was constructed to bolt onto the front of the CI meter, TSM-15. Since the oven has just been completed, no studies of the magnitude of measurement error reduction have been made. This will be included in subsequent reports.

e. Teflon Dielectric Coaxial Cable

For the 50-ohm coaxial cables having a polyethylene dielectric, 50-ohm cables having a Teflon dielectric were substituted. Some improvement was obtained initially. However, the lack of flexibility of the Teflon cables caused the BNC line terminations to become intermittent after a few measurements during which the cables were connected and disconnected each time.

D. Resonator Fabrication and Measurement*

1. Units Fabricated and Measured During the Quarter

During the quarter 70 additional resonators were fabricated. These consisted of

20 each 5th overtone units plated with Cu only
10 each 5th overtone units plated with Al only
10 each 5th overtone units plated with Al and Ag
30 each 9th overtone units plated with Ag only

Of these approximately one-fourth were successfully operated whereas the others did not operate. The parameters of the operable units are displayed in Table I. The high percentage of inoperable units is ascribed to crystals

* Measurements with regard to the temperature cycling studies and the drive level of units are discussed in Section IV F, following.

Table I

Parameters of Operable 100 Mc Resonators*
Fabricated During Period 1 October 1960 - 1 January 1961

Unit Identification	Overtone of Operation	Electrode Metal	Storage Temp(°C)	Test Period (Days)	Series Resistance (ohms)	Total Drift $\Delta f/f$ Parts in 10^7	Calculated Drift rate per month Parts in 10^7
C-5-1	5	Al	60	20	62.0	-55**	83**
C-5-2	5	Al	60	20	31.5	-5.9	8.9
C-5-3	5	Al	60	20	30.5	-2.5	3.8
C-5-5	5	Al	60	20	25.5	-4.0	6.0
C-5-6	5	Al	60	4	30.0	-21.5	540**
C-5-7	5	Al	60	20	24.5	-4.3	6.4
C-5-8	5	Al	60	20	44.5	-	-
D-5-1	5	Al + Ag	60	72	48.5	-4.5	1.9
D-5-3	5	Al + Ag	60	72	40.0	-23.1	9.6
D-5-4	5	Al + Ag	60	72	42.5	-16.3	6.8
C-9-1	9	Ag	60	47	55.5	-6.7	0.45
D-9-1	9	Ag	60	32	56.5	-0.2	0.19
D-9-2	9	Ag	60	32	59.0	-	-
D-9-3	9	Ag	60	32	63.5	-1.2	1.1
D-9-4	9	Ag	60	32	69.0	-1.4	1.3
D-9-5	9	Ag	60	32	64.0	+0.8	0.75
D-9-7	9	Ag	60	32	65.5	-0.2	0.19

* A total of 70 units were fabricated of which only 17 were operable. The high loss included four whole groups of ten each coated with copper or silver. With better blanks and sealing yield is expected to be above 75%.

** Unit leaking.

of the wrong diameter and non-uniform quality. The sealing of the units in glass initially presented a problem which caused a large number of the failures. This problem has since been solved.

Although a large part of the measurement period was interrupted by the necessary modifications to the measuring equipment, measurements were continued when feasible.

Measurements typical of those found are displayed in Figures 3, 4, 5, 6, 7, 8, 9 and 10. It will be noted that measurements for the fifth and 7th overtone units have been much less consistent than those for the 9th overtone units of the last five figures. In fact the units B-9-2, B-9-4 and B-9-6, displayed in Figures 8-10, display no appreciable aging during the period September 16 - January 15, i.e., frequencies have not shown an appreciable directional drift in 120 days and total change has been $<$ a part in 10^7 during that period.

The stability of the ninth overtone units plated with silver has remained outstanding.

2. Resonator Frequency Variation with Temperature

Because of the measurement problem of a resonator displaced at a considerable distance from the bridge a series of measurements was made of a fifth overtone unit mounted directly in the bridge. Measurements were made over a period of several hours as the ambient temperature of the room rose. (Although the building is air conditioned, local temperature control for large assemblages of electronic equipment is inadequate.)

Several days later, after a week-end in which the room became considerably warmer, a new series of measurements was made.

The data obtained are displayed in Figure 11. This revealed the probable continuity of the temperature versus frequency line plot on the second measurement period and that the frequency varied approximately 100 cycles in the range of about 5°C. When the window of the laboratory was opened a drastic upward shift in frequency took place in a short period. The temperature difference effect is quite obvious. Some temperature lag effect between the thermometer and temperature sensitive parts of the circuit are apparent.

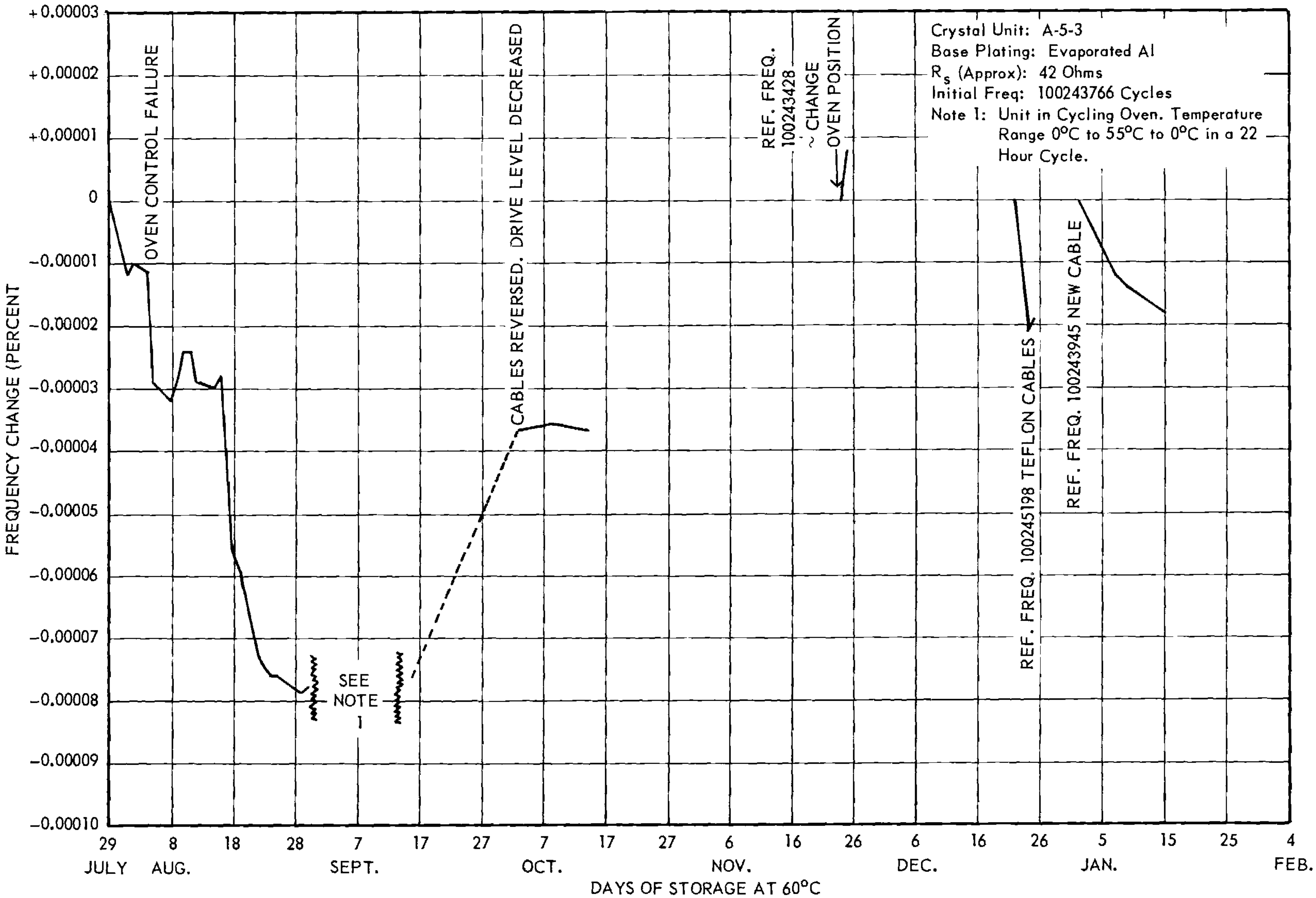


Figure 3. Frequency versus time data for resonator A-5-3, a fifth overtone unit plated with evaporated aluminum.

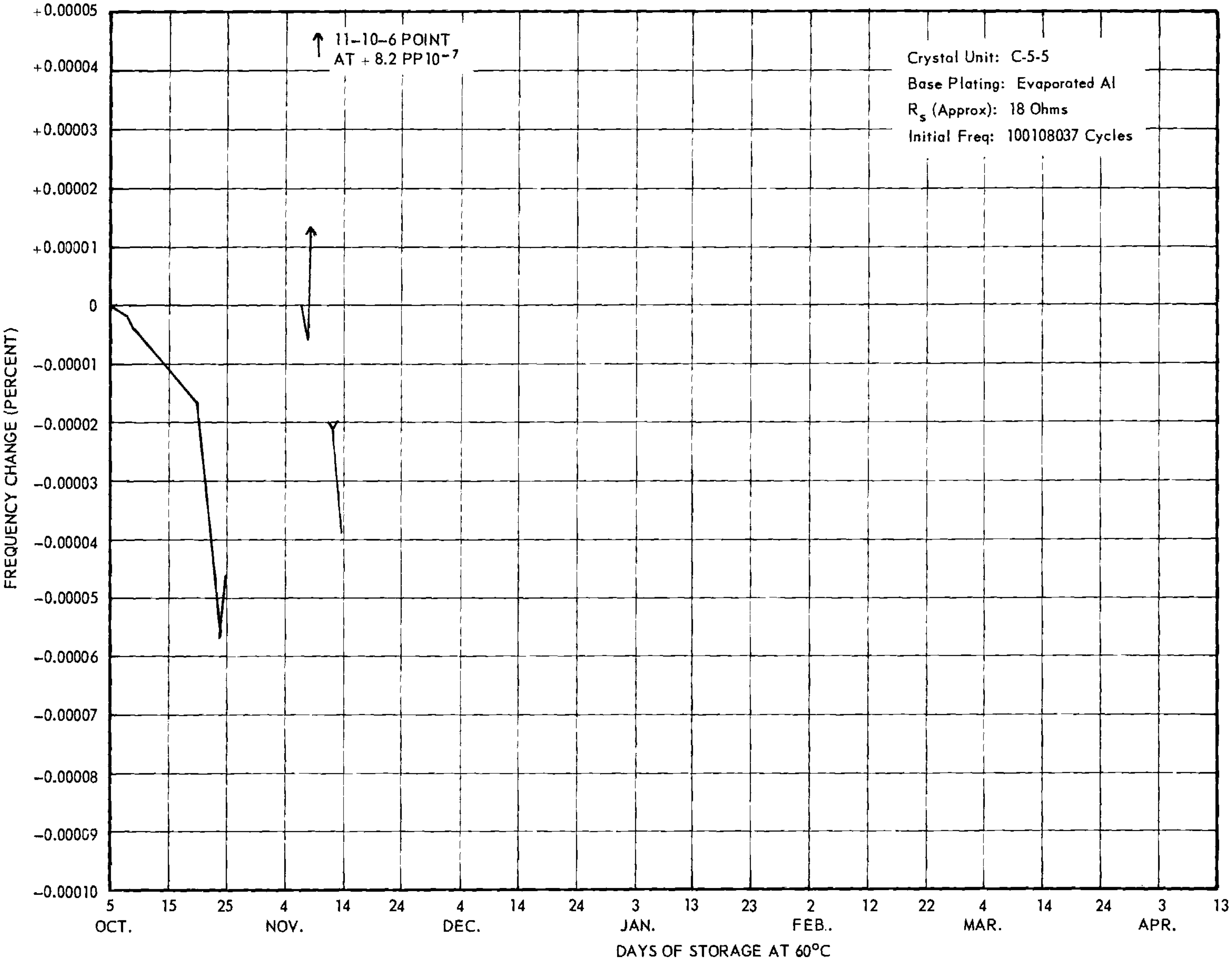


Figure 4. Frequency versus time data for resonator C-5-5, a fifth overtone unit plated with evaporated aluminum.

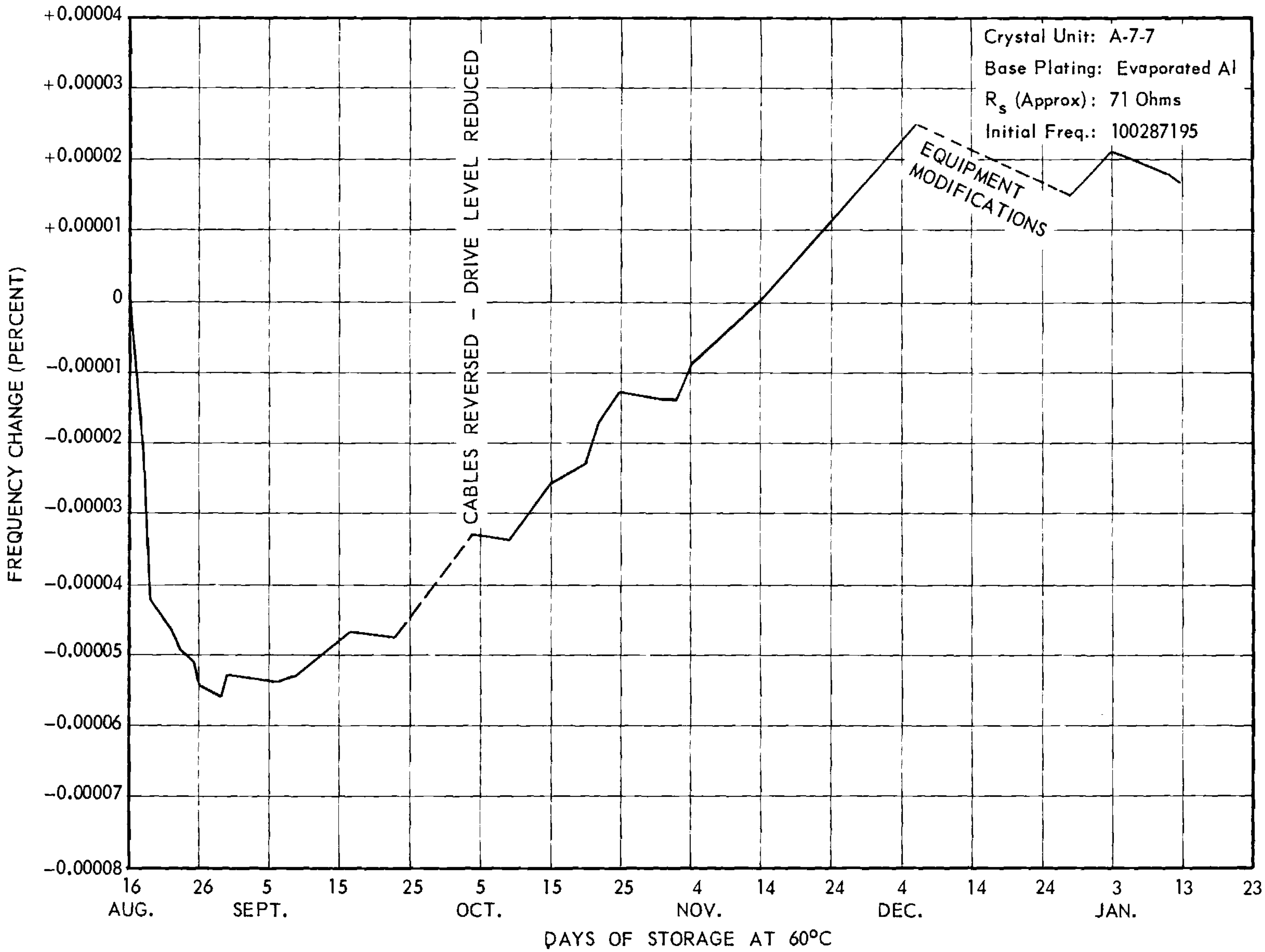


Figure 5. Frequency versus time data for resonator A-7-7, a seventh overtone unit plated with evaporated aluminum.

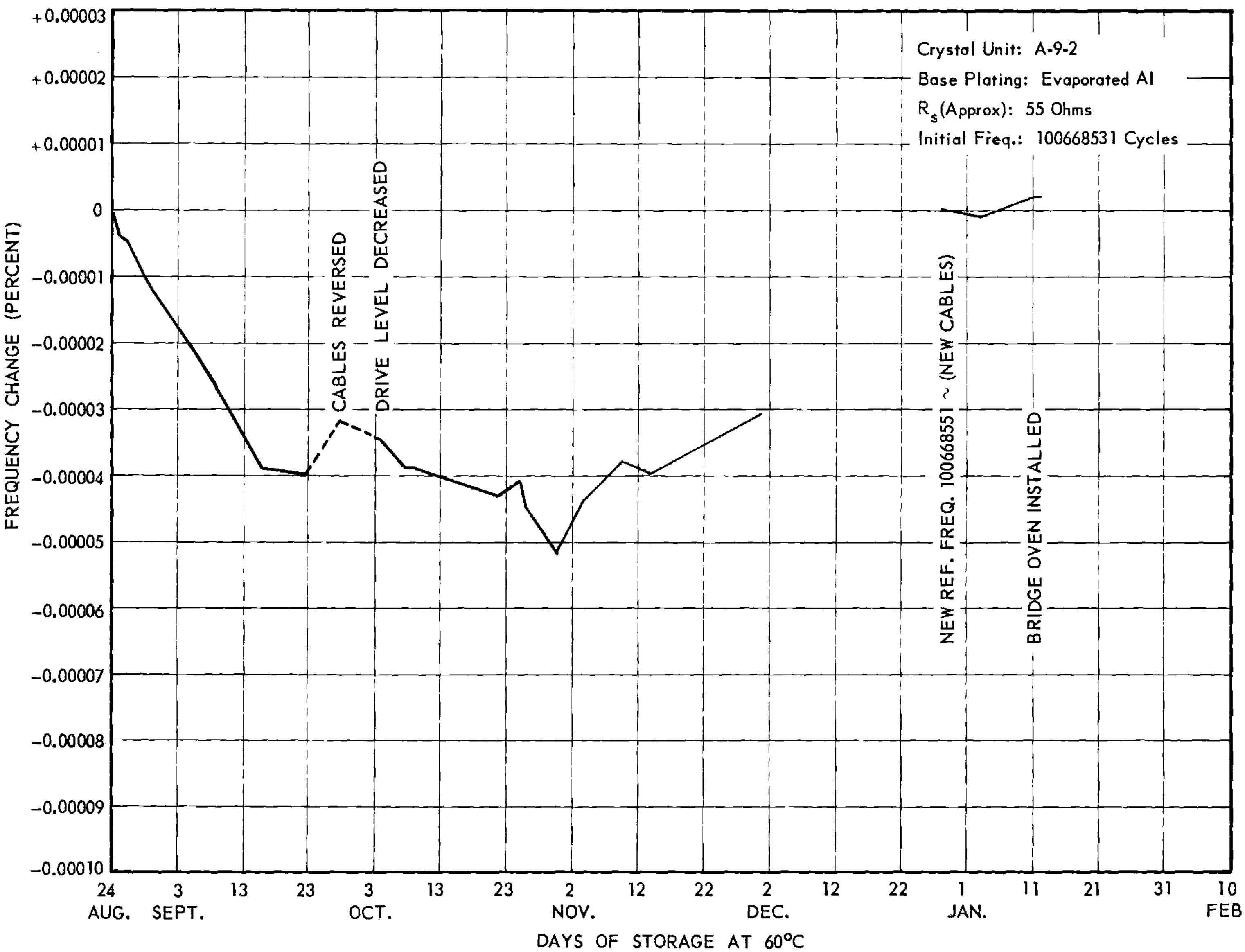


Figure 6. Frequency versus time data for resonator A-9-2, a ninth overtone unit plated with evaporated aluminum.

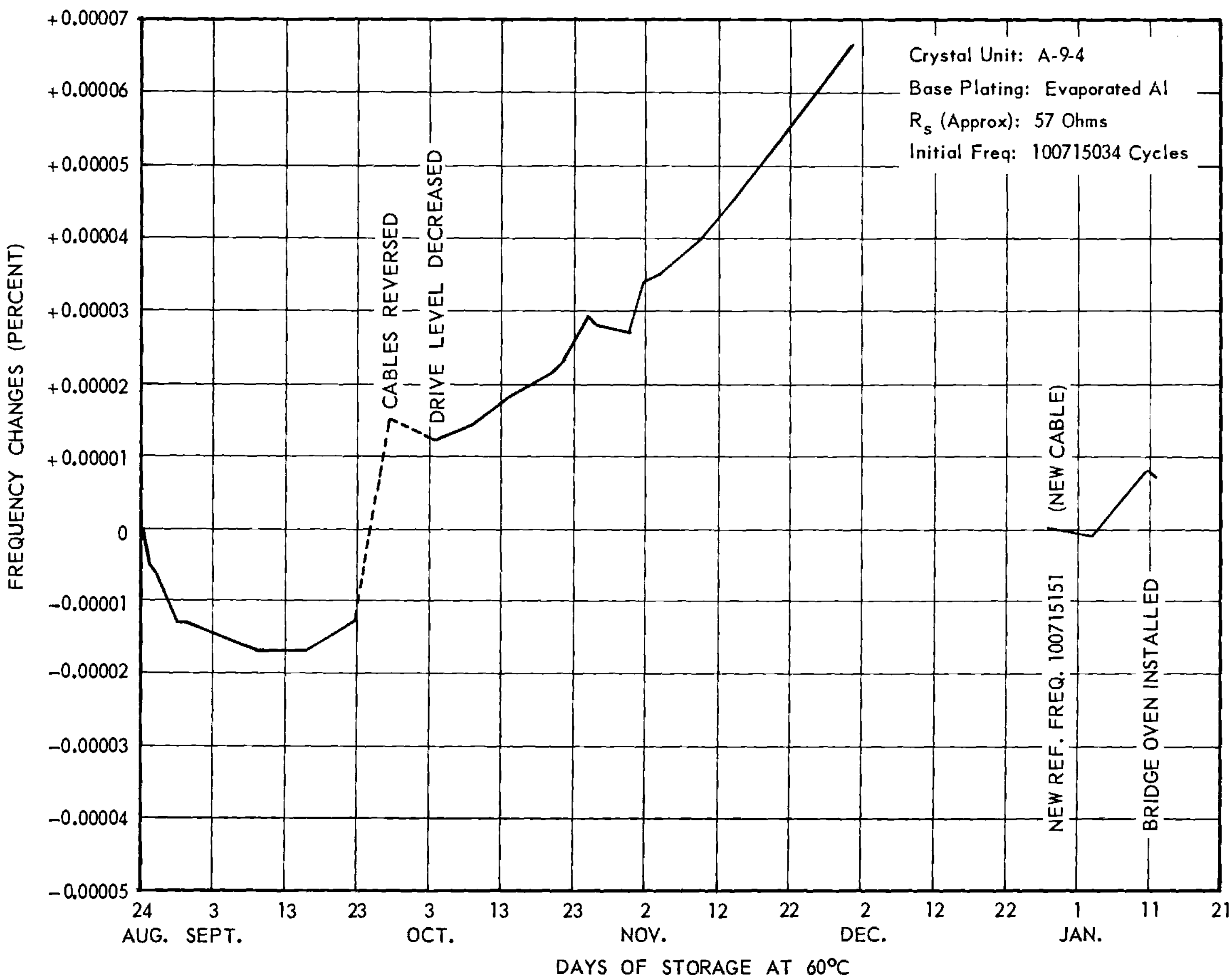


Figure 7. Frequency versus time data for resonator A-9-4, a ninth overtone unit plated with evaporated aluminum.

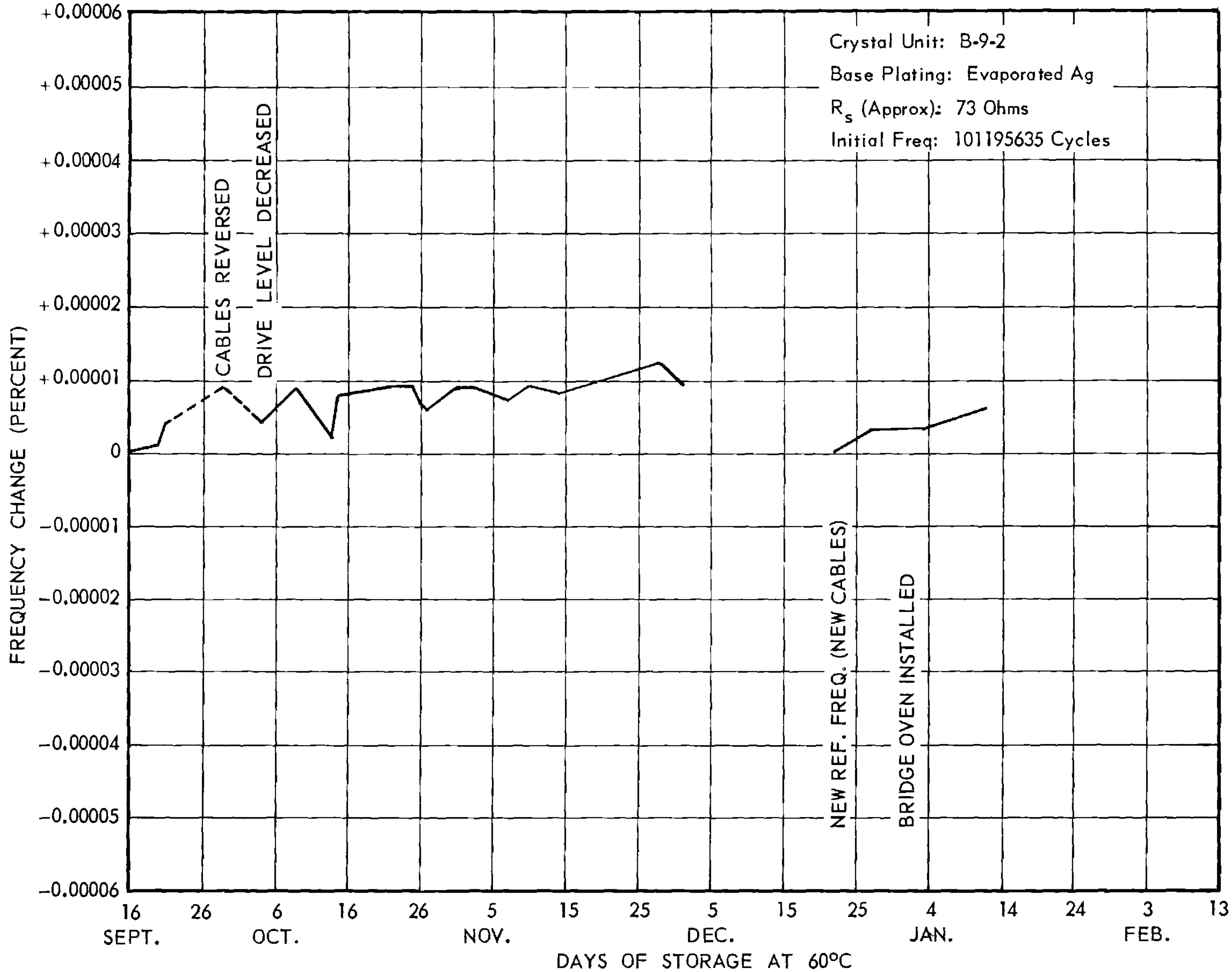


Figure 8. Frequency versus time data for resonator B-9-2, a ninth overtone unit plated with evaporated silver.

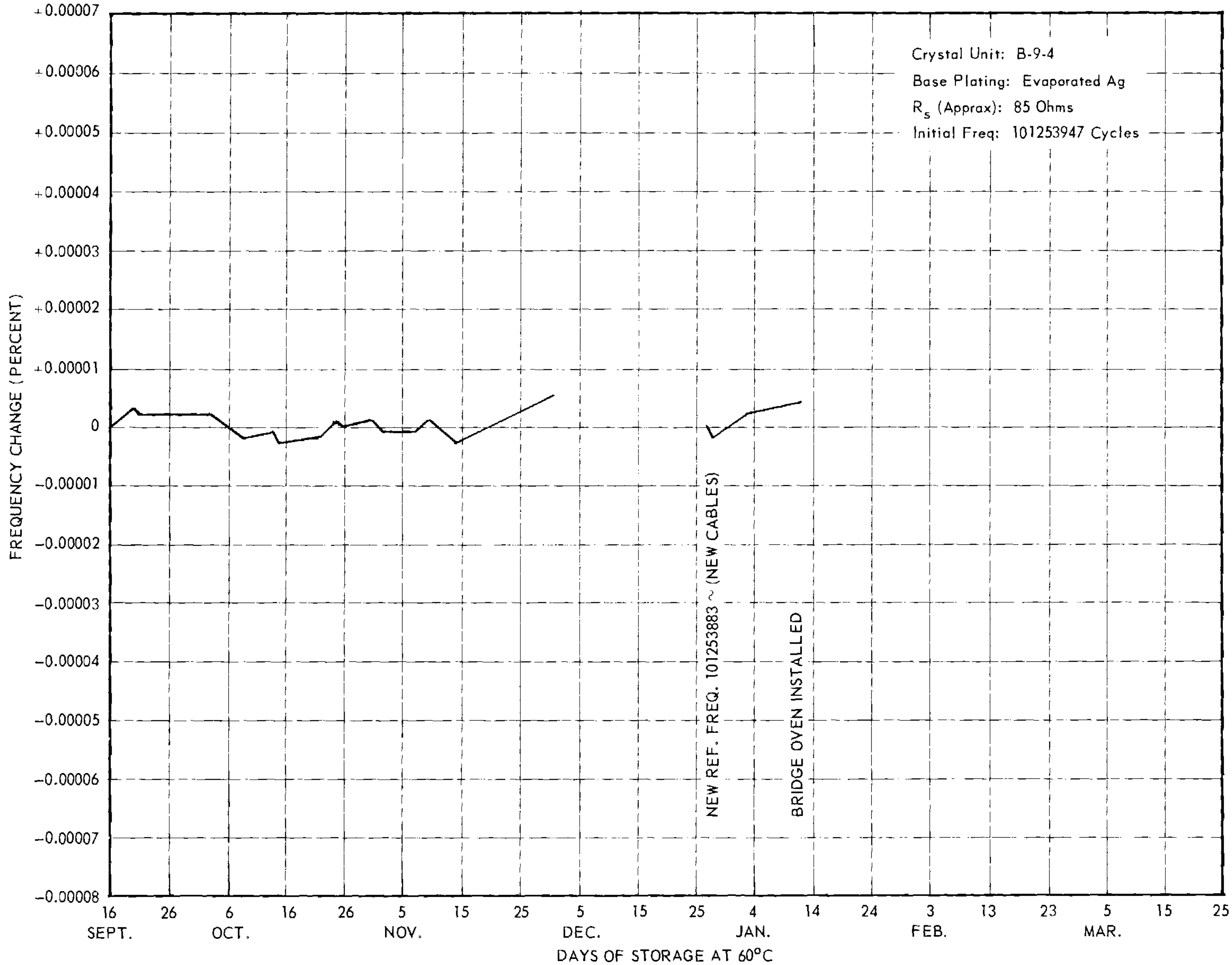


Figure 9. Frequency versus time data for resonator B-9-4, a ninth overtone unit plated with evaporated silver.

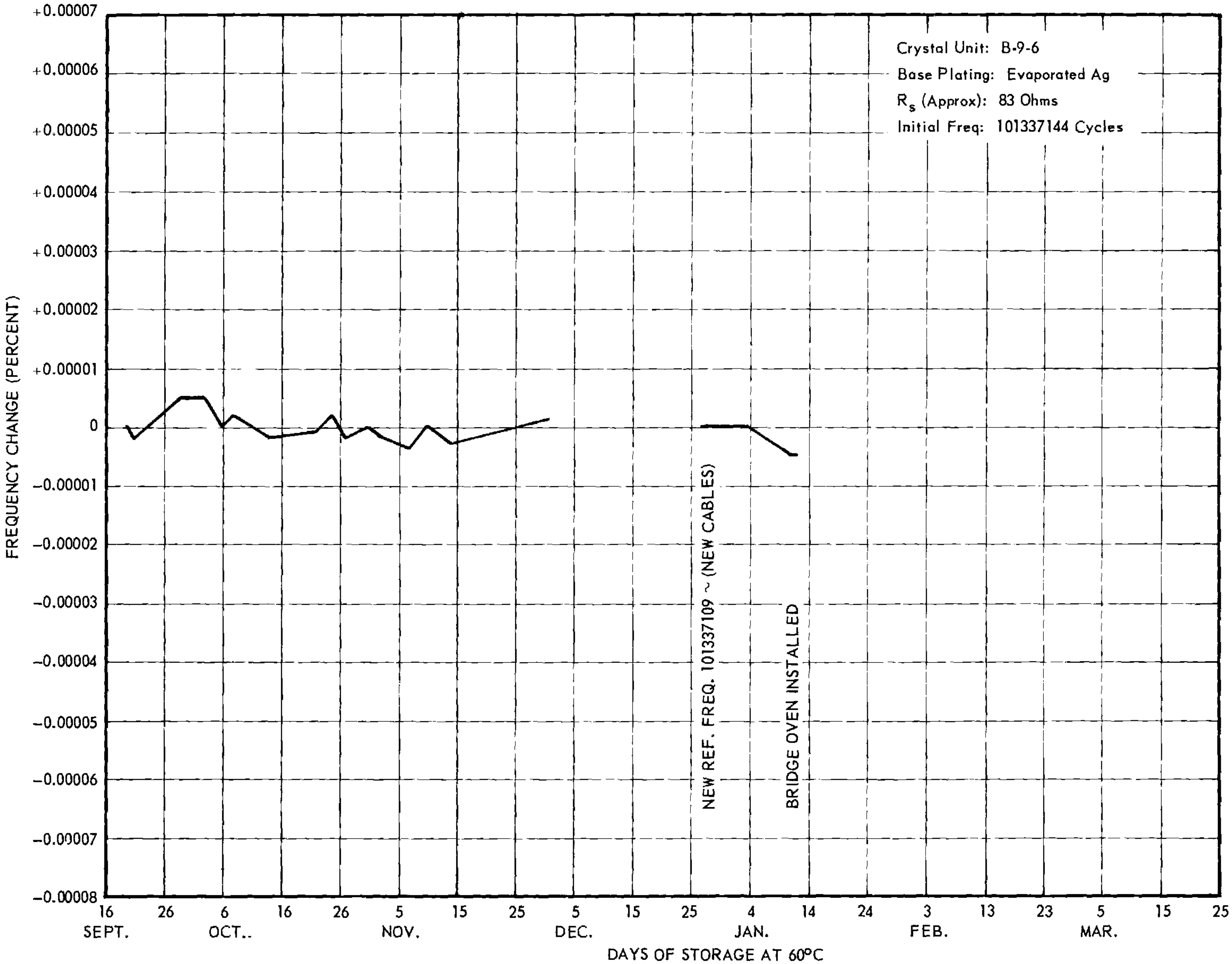


Figure 10. Frequency versus time data for resonator B-9-6, a ninth overtone unit plated with evaporated silver.

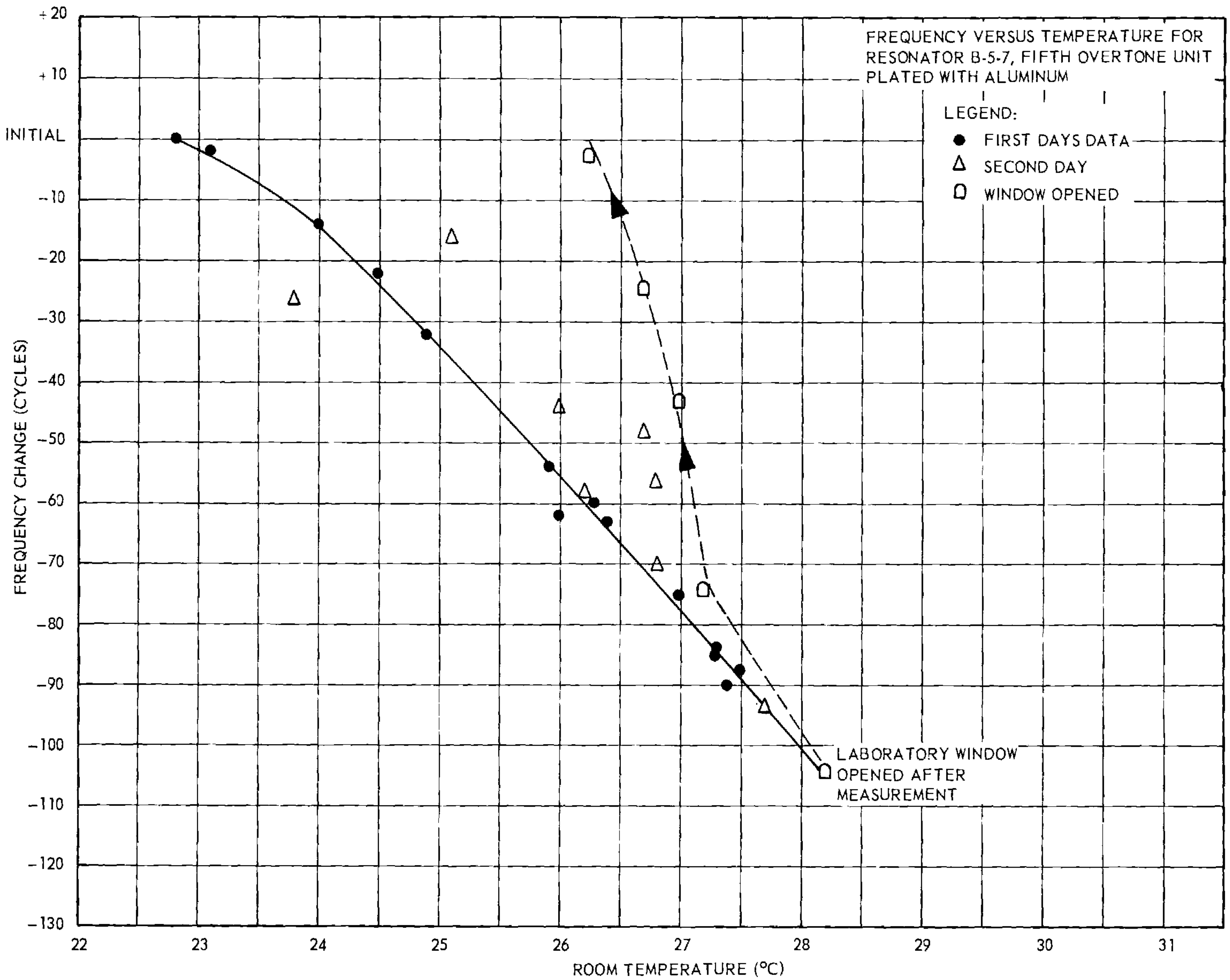


Figure 11. Frequency versus temperature data for resonator B-5-7, operated at room temperature and measured on two different dates.

These measurements emphasized the need for better temperature control of the room, the bridge, and the line, in addition to control of the crystal temperature to permit frequency measurements accurate to a few parts in 10^8.

It was anticipated that temperature-versus-frequency measurements for a unit made over a long period of time would reveal aging over the time interval in case of measurement difficulty at the oven site or that each unit might be mounted in an independent oven on the bridge. Fortunately, however, the measurement problem has now been resolved without resorting to such methods.

E. Studies of the Effect of Radiation

Plans for studies of the effect of radiation on the 100 Mc resonators have been made. However, delays in achieving accurate measurement methods have nullified further progress in radiation studies except on an excessively coarse scale.

Frequency measurement methods have now progressed to a satisfactory stage and effects of radiation damage will be examined during the succeeding Quarter.

F. Comments

1. General

Measurement difficulties during a large portion of the quarter interfered with the aging studies. However, these have now been largely overcome by a thorough investigation of potential measurement techniques*. Currently two techniques have been established which appear to give accurate results to $\pm$ 1 part in 10^8. These are the old VHF bridge system, with better connectors and a temperature stabilized bridge, and the capacitance bridge described in Section IV, C, 2. These two bridges allow independent measurements of the same resonator by two systems; this, in turn, offers a check as to whether measurements by each describe the aging of the resonator as being in the same direction and of the same magnitude and rate.

* This conclusion is based on measurements made subsequent to the reporting period.

2. Temperature Cycling of Resonators

The crystal units temperature cycled between 0°C and 60°C have not been measured in the cycling oven. The heating and cooling rates of this oven were intentionally made large to allow proper tracking of the oven temperature with the cycle control thermostat. Thus even when the cycle control motor is off the oven temperature cycles about a given value too rapidly for accurate frequency measurement.

The measurement technique employed has been to first establish the aging rate of a unit at a fixed temperature such as 0°C or 60°C, place the crystal in the cycling oven for a period of time and then return it to the fixed temperature oven for a determination of frequency and aging rate changes.

3. Measurement of Resonator Drive Level

Crystal drive level was not measured directly as a RF voltage drop across the crystal or as RF current through the crystal. Rather, RF voltage is monitored at the CI meter across the crystal contact terminals.

Consider Figure 12 which originally appeared as Figure 108 in the final report of Contract No. DA-36-039 SC-78905 and is repeated here for convenience.

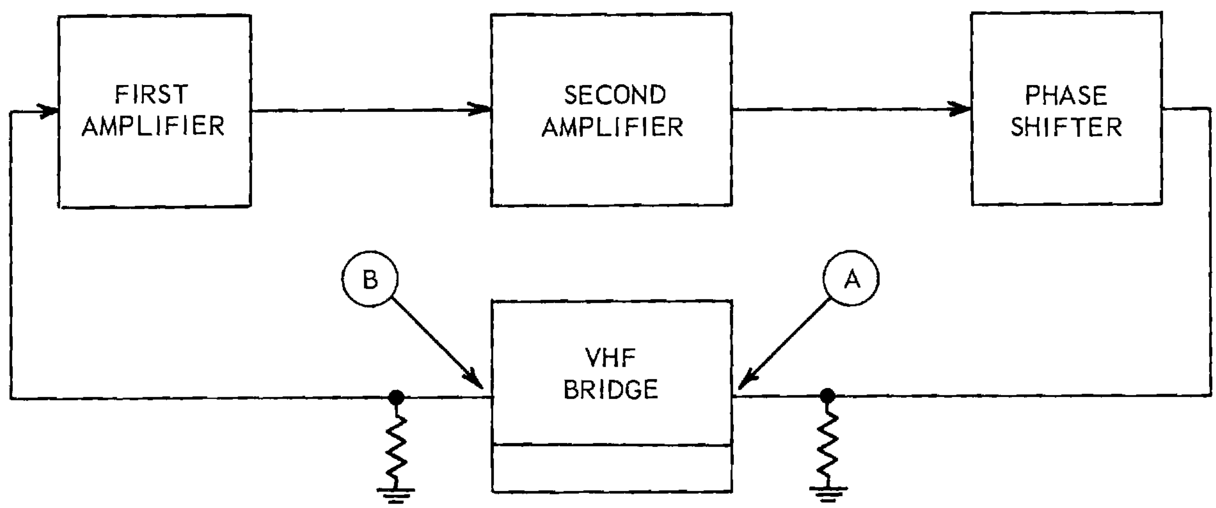


Figure 12. Functional Schematic Diagram of Oscillator and VHF Bridge.

Points A and B are the crystal terminals of the CI meter (Model 2, Serial No. 3 from Contract DA-36-039 SC-56730). The RF voltage to ground at point A minus the voltage at point B would give the voltage across the bridge. The latter voltage has about one half the voltage at point A depending upon the particular crystal being measured. Assuming a crystal of 100 ohms resistance and the bridge at balance, about one half of the total bridge voltage appears across the crystal. Assuming 25 millivolts at point A, the bridge voltage would be 12.5 mv and the crystal voltage (for a 100 ohm crystal) 6.25 mv. The crystal power would then be:

$$(1) \quad P_c = \frac{E^2}{R_s} = \frac{(6.25 \times 10^{-3})^2}{100} = .39 \text{ microwatts}$$

It was found to be inconvenient to measure both the voltage at points A and B while balancing the bridge. The assumption was made that at balance the bridge impedance would be a constant as would the impedance from point B to ground. Thus only the voltage at point A was monitored. This latter voltage was normally held at 25 mv by manual operation of the screen voltage control.*

* This voltage was subsequently dropped to only 10 mv after installation of automatic drive level control of the oscillator circuit. Prior to this all units would not operate at so low a level.

V. CONCLUSIONS

The VHF bridge frequency measurement system used, during the initial quarter, was of inadequate accuracy for frequency measurements better than approximately $\pm$ 5 parts in 10^8. Temperature control of the bridge itself and refinement of the coaxial lines and connectors, have improved this bridge to the point that resettability approaches a desired accuracy of $\pm$ 1 part in 10^8.

A second VHF bridge consisting of a capacitance bridge, isolated from the CI Meter Oscillator by a Ferrite transformer, and permitting oscillation of the crystal with one side connected to ground, offers a similar accuracy; it also offers a simple measurement for determining drive level. These two methods are expected to solve the measurement dilemma.

Aging measurements were interfered with by the experiments on measurement technique, but several units operating over a period of 120 days have continued to show little or no aging, i.e., no directional drifts and changes of less than a part in 10^7 in 120 days.

VI. PROGRAM FOR THE NEXT INTERVAL

Improvements in the frequency measuring system and the supply of quartz crystal blanks make it possible for the next quarter to place major emphasis on the fabrication and measurement of the variously coated resonators. Of primary interest will be those plated with silver or aluminum and overplated to frequency with the same metal. Measurements of the Q of the crystals will be undertaken.

Initial studies of the effects of radiation on aging will be made.

Measurements of the aging of units already fabricated will be continued.

VII. IDENTIFICATION OF KEY TECHNICAL PERSONNEL

The following persons have been employed on this project during its second quarter for the times indicated.

Name	Position	Time (Hours)
Richard B. Belser	Project Director	216
Douglas W. Robertson	Research Engineer	8
W. Bruce Warren	Research Engineer	32
Samuel N. Witt	Research Engineer	70
Walter H. Hicklin	Ass't. Research Engineer	487
James O. Darnell	Research Assistant	468
Carol M. Shirley	Technician	454
W. Donald Dawson	Student Assistant	64

Mr. Belser has been associated with resonator aging studies sponsored by USASRADL for over ten years and has been assisted by Mr. Hicklin for approximately nine years. Mr. Robertson, Mr. Warren and Mr. Witt, electrical engineering graduates of Georgia Tech with M.S. degrees, have served in the capacity of project directors on projects sponsored by both Signal Corps and Air Force Agencies for a number of years and each has had recent industrial experience with leading communication manufacturers.

Respectfully submitted,

Richard B. Belser
Project Director

Approved:

Vernon Crawford
Head, Physics Branch
Physical Sciences Division

Report No. 2 (Second Quarterly), Project No. A-508

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Stability Studies of Quartz Crystals for Satellites

By
R. B. Belser and W. H. Hicklin

Report No. 3 (Third Quarterly)
Contract No. DA-36-039-sc-85363
1 January 1961 to 1 April 1961
Georgia Tech Project No. A-508

Placed by the U. S. Army
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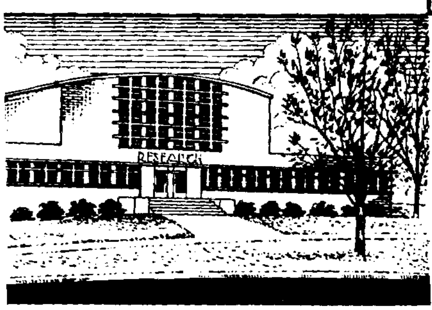

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I. PURPOSE

The purpose of this project is to develop AT-Cut quartz resonators of 100 Mc frequency with stabilities suitable for operation in satellites or other space vehicles. Initial target stabilities are a maximum frequency deviation of ± 0.3 parts per million per year and ultimate desired stabilities of ± 0.1 parts per million per year. These stabilities are to be maintained while the resonator is subjected to temperature cycles of 0° to 60°C one or more times per day and exposed to radiation similar to that of the Van Allen belt.

Resonators operated in the 5th, 7th, and 9th overtone modes will be investigated. Plating metals are to be silver or aluminum. Effects of radiation similar to that in the Van Allen belt on the stability of the 100 Mc resonators will be studied.

II. ABSTRACT

During the third quarter the frequency measurement problem was resolved. By the adoption of a single-ended capacitance bridge and the application of automatic gain control to the driving oscillator frequency measurements to within $\pm$ 2 parts in 10^8 were obtained.

Fifty, fifth-overtone, 100 Mc resonators were fabricated during the quarter. A batch of poor bonding cement and a number of leaking glass stems reduced the yield of operable units to 27. Correction of the bonding and the glass stem problems increased the yield to 80 percent during the latter portion of the quarter.

Selected 100 Mc resonators, aged at 60°, have continued to show minimal aging of less than 1 part in 10^7 in six months; and very low aging has been experienced at 0°C for similarly fabricated units.

Twelve resonators were exposed to radiation from a 12,000 curie Cesium-137 source and two to 1 Mev protons from a Van de Graaf Accelerator. The units exposed at high intensities (1.4×10^6 rad/hr) to the Cesium source for 10 to 30 minutes shifted downwards, in accordance with time of exposure, up to 10 ppm. Aluminum plated resonators exhibited somewhat greater shifts than silver plated ones. Resonators exposed for one hour at 2.0×10^4 rad/hr, approximately equivalent to the intensity of the Van Allen Belt, shifted downward an average of only 20 cycles.

Two resonators exposed to the Van de Graaf proton beam for one second shifted one and 33 cycles respectively.

The more severely irradiated units aged upwards at rates of about 2 to 10 parts in 10^8 per day, initially, but the rate of change was decreasing at the end of 30 days. Units undergoing lesser downward shifts exhibited lower recovery rates.

III. PUBLICATIONS, LECTURES, REPORTS, AND CONFERENCES

No publications or lectures were made during the third quarter. Monthly letter reports and Report No. 2 (Second Quarterly) were completed.

Mr. P. E. Mulvihill, Project Engineer, visited the Georgia Institute of Technology on 6 March 1961. The progress and work of the project were examined and discussed. The radiation facilities were visited. Tentative plans for increased oven capacity for aging studies of commercially fabricated units at 85°C were described and considered. No action was taken on this matter pending further instructions.

IV. FACTUAL DATA

A. Introduction

The principal apparatus for fabrication, storage, and measurement of the 100 mc quartz resonators under study have been described in the preceding Quarterly Reports (Nos. 1 and 2).

Modifications made to the measurement apparatus, the principal problem thus far, have resulted in the adoption of the single-ended, capacitance bridge described on pages 6 and 7 (Figure 1) of Quarterly Report (No. 2). In addition, automatic gain control has been applied to the crystal drive oscillator. These two improvements have brought frequency measurements to the desired accuracy of $\pm$ 2 parts in 10^8.

The necessary facilities and personnel for performing the radiation damage studies of the 100 mc resonators have been made available and the results of the initial experiments are reported.

B. Apparatus and Procedures

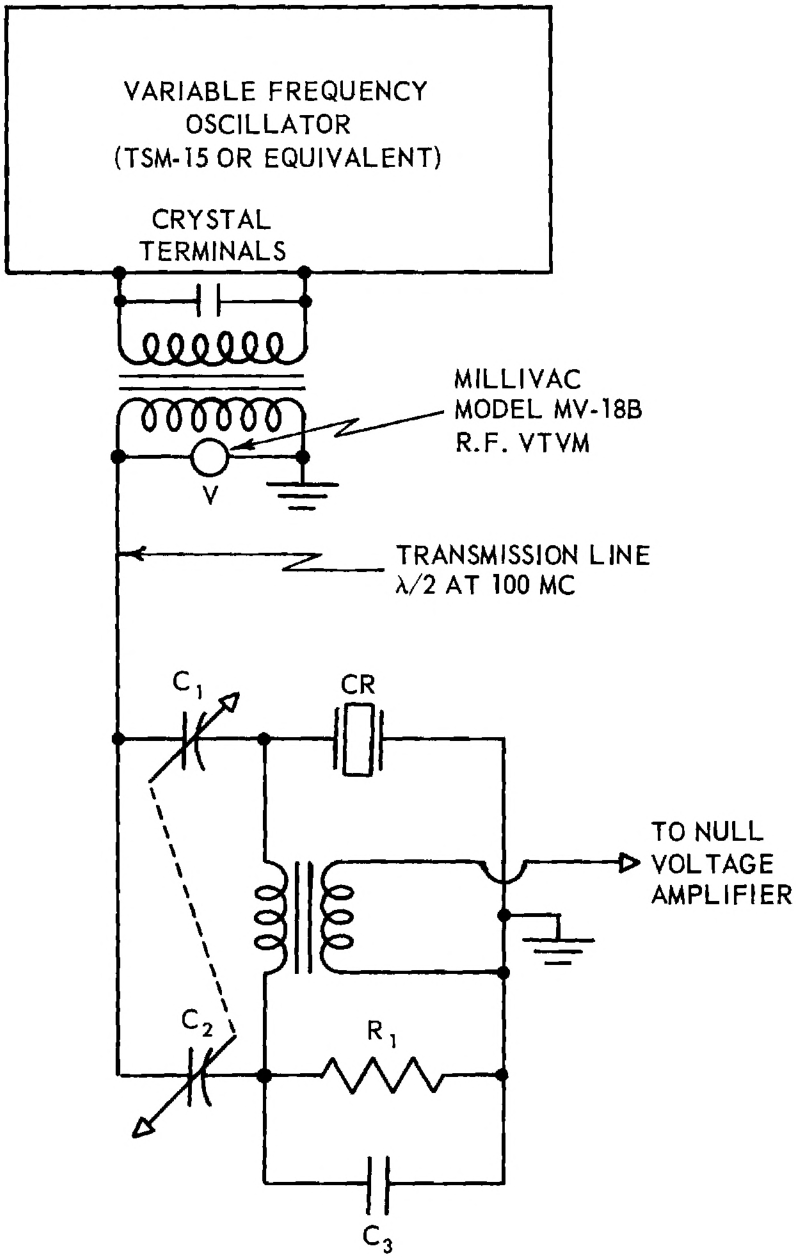
1. Measuring Equipment

a. The single-ended capacitance bridge

During the present reporting period the change-over to the single-ended bridge circuit was completed. The circuit used is shown in Figure 1. This arrangement has proven to be superior to the previous system. The improvement is due primarily to the fact that the crystal loop does not incorporate resonant lines, the characteristics of which can vary with time, temperature, and humidity.

b. Oscillator automatic gain control

An automatic gain control (AGC) circuit was designed and constructed for controlling the oscillator screen voltage. The circuit is shown in block diagram form in Figure 2 (A). The schematic of the control stage is shown in Figure 2 (B). The following advantages are obtained by operation with AGC.



T_1 – FERRITE TRANSFORMER (UNITY TURNS RATIO)
C_1, C_2 – DIFFERENTIAL CAPACITOR (3–20 $\mu\mu f$)
R_1 – RESISTOR (100Ω)
CR – CRYSTAL RESONATOR
C_3 – COMPENSATING CAPACITOR (ABOUT 10 $\mu\mu f$)

Figure 1. Single-ended bridge for improved measurement accuracy at high frequencies.

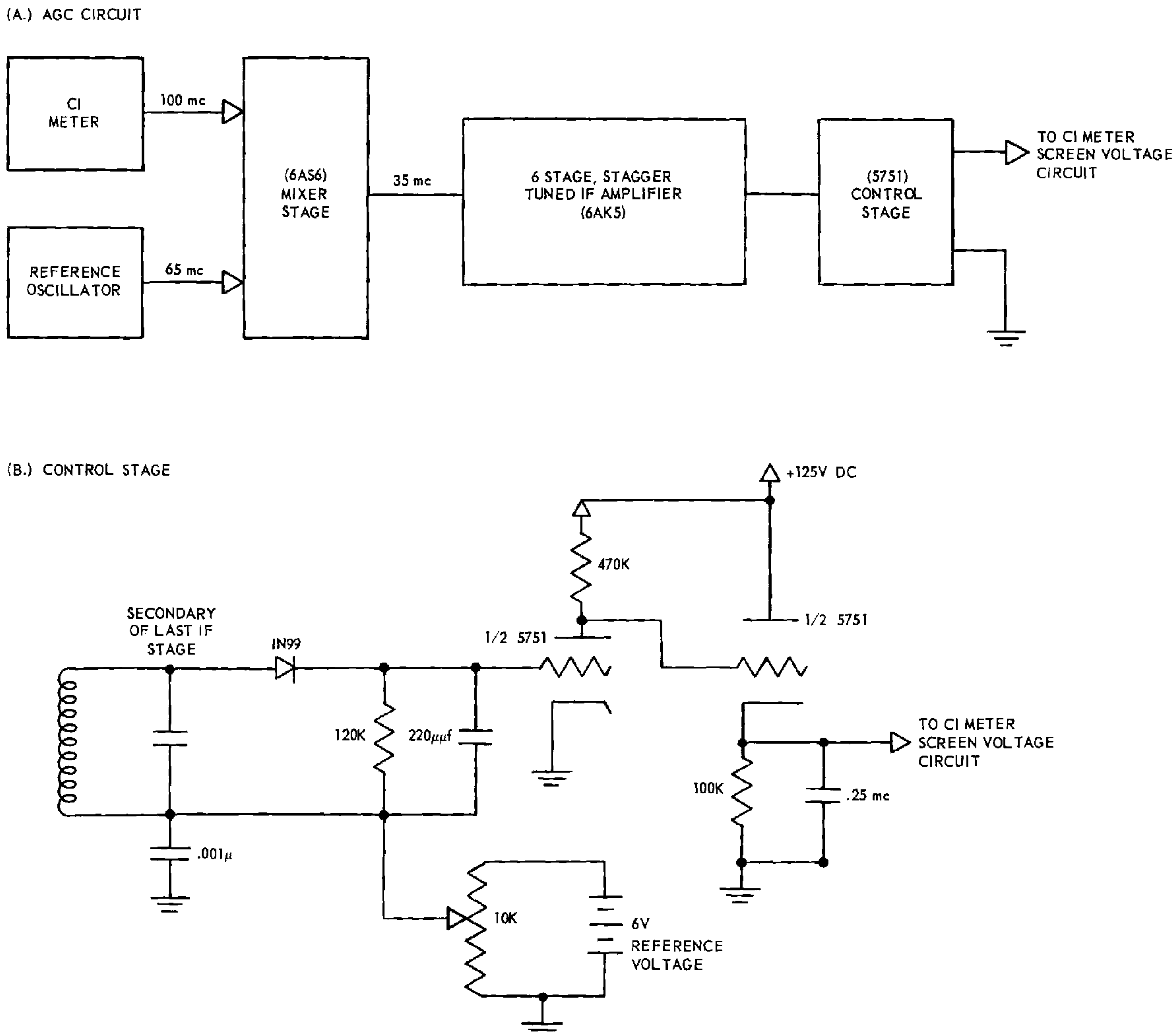


Figure 2. Block diagram of the AGC circuit used on the bridge driving oscillator and circuit of last stage.

(1) Lower crystal drive levels may be used.

(2) The danger of overdriving the crystals is reduced.

(3) Less time is required to complete a measurement since the operator does not need to "ride gain" on the drive level control.

2. Final Plating of VHF Units

100 mc crystal resonators operating at the higher overtones usually have a considerable frequency spread after base plating. Part of the spread is due to the lack of an exact numerical relation between the fundamental and overtone frequency. Thus, plate-back during final plating may be considerable. It has been found that the 5th mode crystal units may be plated back as much as .5 mc without loss of activity, i.e., Rs increases, provided the crystals are plated uniformly on each side at the same time. Also, plating should cover the base plate spot as uniformly as possible.

Some initial difficulty was encountered obtaining uniform evaporation from apparently identical filaments operated either series or parallel. The metal to be evaporated is not uniformly distributed over the separate filaments and, having generally a much lower resistance than the filament, represents a partial short circuit; this causes the power dissipation of the two filaments to be dissimilar. The circuit of Figure 3 was tried and found to be of considerable benefit in providing uniform evaporation from each filament.

The operation is as follows:

a. Set T_1 and T_3 to zero volts.

b. Load pre-formed filaments.

c. Close system and evacuate.

d. Close switch S_1.

e. Increase output of T_1 until load on filament A just melts.

f. Increase output of T_3 until load on filament B just melts. If the power handling capacity of the variable transformer T_1 is sufficient no interaction between controls is obtained.

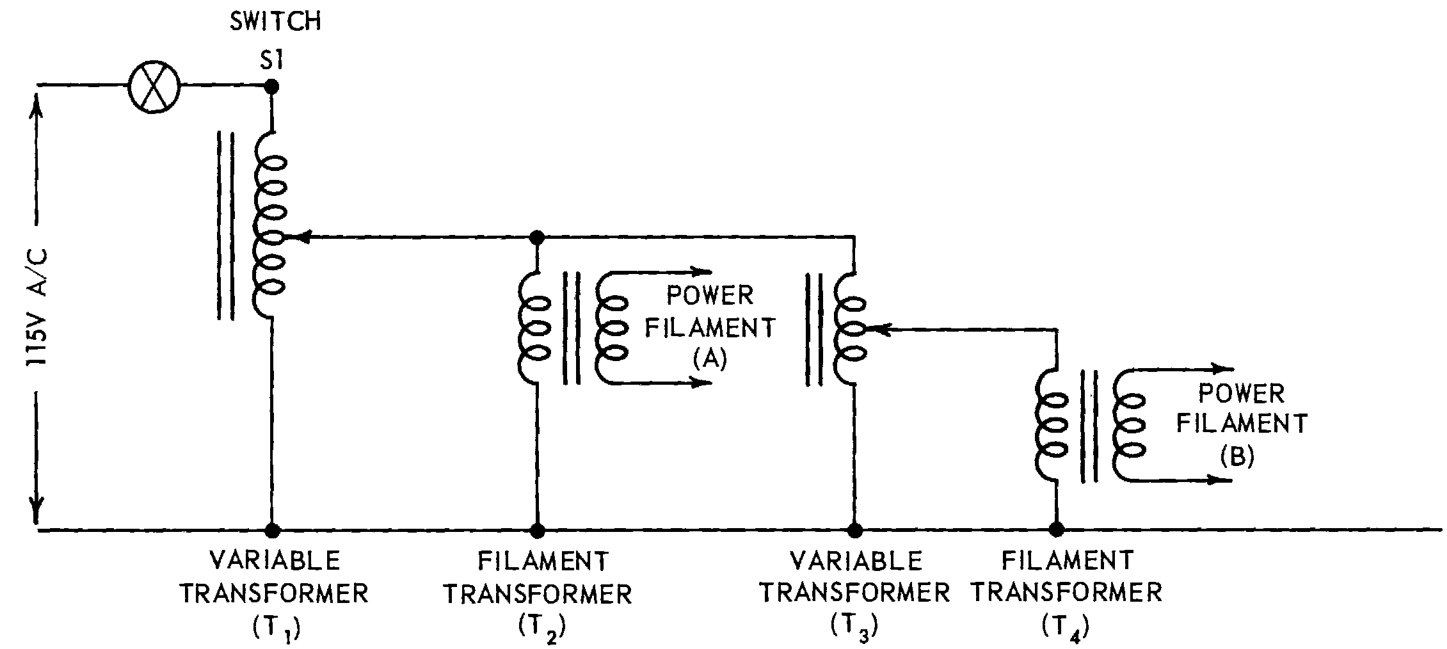


Figure 3. Circuit used to maintain uniform evaporation from two filaments operated simultaneously during final plating to frequency.

g. With both filaments pre-set to operate at the same temperature, the temperature may be varied above and below that temperature by the operation of T_1 alone.

3. Radiation Damage Study Facilities*

a. Introduction

One of the purposes of this research project is to investigate the effects of radiation similar to that in the Van Allen belt on the stability of the AT-Cut quartz resonators of 100 mc frequency. In 1959 at the first nationally sponsored Symposium on the Exploration of Space, Van Allen** presented data representing intensity values in the Van Allen belt:

1. Inner zone - 3600 km on the geomagnetic equator
   a. Electrons of energy greater than 20 Kev: maximum unidirectional intensity of 2 x 10^9/cm^2-sec-ster
   b. Electrons of energy greater than 600 Kev: maximum unidirectional intensity of 1 x 10^7/cm^2-sec-ster
   c. Protons of energy greater than 40 Mev: omnidirectional intensity of 2 x 10^4/cm^2-sec.
2. Outer zone - 16,000 km on the geomagnetic equator
   a. Electrons of energy greater than 20 Kev: omnidirectional intensity of 1 x 10^{11}/cm^2-sec
   b. Electrons of energy greater than 200 Kev: omnidirectional intensity of 1 x 10^8/cm^2-sec
   c. Protons of energy greater than 60 Mev: Omnidirectional intensity of 10^2/cm^2-sec.
   d. Protons of energy less than 30 Mev: No significant data.

* This section contributed by R. C. Palmer, Radio Isotopes Laboratory of the Engineering Experiment Station, Georgia Institute of Technology.

** J. A. Van Allen, J. Geophy. Res 64, 1683(1959).

From Range - Energy curves for charged particles*, it can be shown that the glass envelope of the resonator will stop all incident protons of energy less than ~12 Mev and all incident electrons of energy less than ~800 Kev. All incident particles of higher energy will have their energy degraded by the envelope with the energy after penetration dependent on the incident energy and the thickness of the envelope. The effects of incident particles, however, are only part of the total effects which will be observed. Further radiation of the quartz crystal will result from secondary electrons, bremsstrahlung, gamma rays, mesons, and neutrons produced from the magnitude of nuclear reactions that high energy protons can initiate.

An investigation of the effects of this radiation would in the first instance be highly qualitative in nature as the only source of such varied particles is the Van Allen belt itself. The effect to be produced by a particular type of radiation can, however, be studied quantitatively as well as qualitatively; the information gained can then serve as the basis for prediction and for investigation of the effects of the Van Allen belt on the resonator.

b. Irradiation Facilities

The radiation sources available for this work at the Georgia Institute of Technology are: (1) a 12,000 Curie Cesium-137 Research Irradiator, and (2) a 1 Mev Van de Graaf Accelerator.

1. The Cesium-137 Research Irradiator**

The Cesium-137 Research Irradiator consists of twelve 5/8-inch diameter brass tubes surrounding a 1-14/16-inch brass tube. Around the 5/8-inch tubes are twelve additional 1-inch diameter brass tubes. This tube bundle extends 8.5 feet into the ground. Twelve Cesium-137 sources (approximately 1000 curies each) are contained in the 5/8-inch diameter tubes at a depth of 8 feet below floor level. Samples to be irradiated are

* W. A. Aron, B. G. Hoffman, and F. C. Williams, AECU-663 (1951).

** R. C. Palmer and R. W. Carter, Int. J. Applied Rad. and Isotopes 9, 123 (1961).

lowered into either the center tube or one of twelve outside tubes. Figure 4 shows the dose variance with position in the irradiator. By varying the position of the sample any dose rate from 1.4×10^6 rad/hr to ~10 m rad/hr (not shown) can be obtained. This range is such that any "total dose rate" of the Van Allen belt can be achieved.

2. The Van de Graaf Accelerator

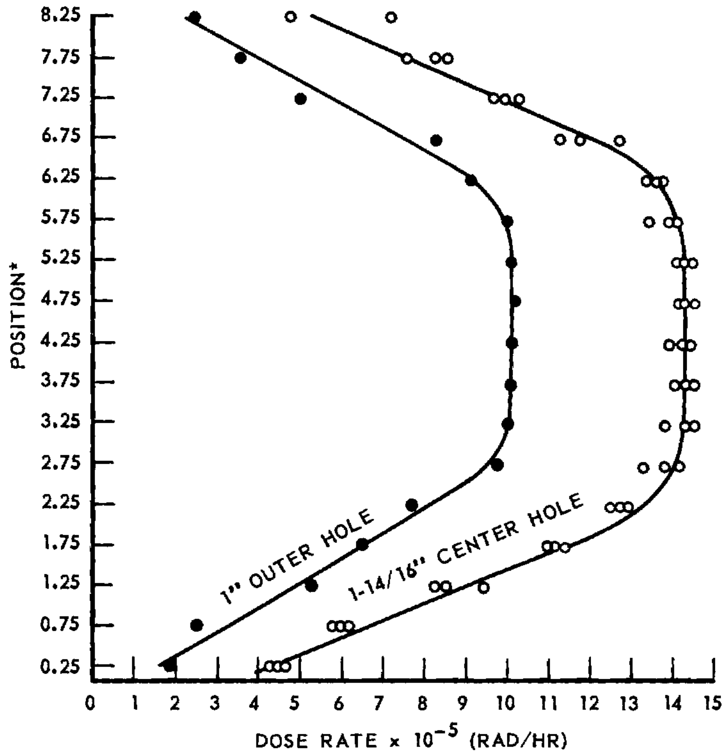
The Van de Graaf Accelerator, which is housed in a specially designed facility in the Radioisotopes Laboratory, is a 1 Mev positive-ion accelerator. It contains a positive-ion source that can provide a continuous ion current of up to 70 micro-amperes. With hydrogen as the source gas, this current is about two-thirds protons (H^+ ions) and one-third molecular ions (H_2^+). We are currently using a mixture of ordinary and heavy hydrogen (deuterium) so that the beam contains about 25 micro-amperes each of protons and deuterons, together with lesser amounts of the molecular ions H_2^+, HD^+, and D_2^+.

The beam emerges vertically from the bottom of the machine and passes through an evacuated tube into the target room below, where it is deflected 90° into a horizontal path by a large electromagnet. By varying the magnetic field we can direct to the exit portal at will either the mass-1 beam of protons or the mass-2 beam of deuterons (and H_2^+ ions). The magnet can be rotated about a vertical axis, so that the emerging beam can be directed to any azimuth. It may be directed through any of several ports in the target-room wall into the adjacent physics laboratory.

c. Experimental Work

1. Introduction

During the quarter fifty 100 Mc resonators have been fabricated. Of these only 27 were successfully operated. The high incidence of failures is ascribed to troubles with bonding cements, overcoating difficulties, and stem leakage. These are described and discussed in subsequent sections.



* The position is measured upward in inches from the bottom of the sample space within the sample carriers.

Figure 4. Dose variance with vertical movement in the Cesium-137 gamma radiation source.

Measurements of older units were continued and initial studies of the effects of radiation exposure in the frequencies of the quartz resonators are described.

2. Resonators Fabricated and Measured During Quarter

Resonators fabricated during the quarter were predominantly of the fifth overtone type. The 27 operable units were all of the fifth overtone type. Fifteen units were base plated with silver only, three with copper only and nine with silver plus silver. The parameters of these units are outlined in Table I.

Nineteen of these units were stored in the 60°C oven and measured for periods up to 84 days. Eight of the units were stored in the 0°C oven and have been measured for a period up to 13 days.

The best unit exhibited in the table (F-5-1-Cu) shows a drift of only +7 parts in 10^8 during a period of 60 days. The R_s of this particular unit, base plated only with copper, was 39 ohms. The data for this unit are shown in Figure 5. A similarly excellent behavior was exhibited by unit F-5-4-Cu as shown in Figure 6.

Note that both of these resonators exhibited positive aging.

3. Resonators Fabricated Previously and Continued on Measurement

The ninth overtone resonators which have now been in operation for periods of more than 200 days have continued to show exceptional stability.

Some of these units have been selected for storage in the temperature cycling ovens, storage at 0°C and exposure to radiation. These will be discussed in paragraphs devoted to these particular studies.

4. Resonators Temperature Cycled over the Range 0°C to 60°C

Although a few units have been temperature cycled at a uniform rate through the temperature range 0°C to 60°C through one complete cycle each 24 hours during the work thus far, frequency measurement difficulties and the radiation study experiments diverted the major effort from this area during the current quarter.

TABLE I

Parameters of Operable 100 Mc Resonators
Fabricated During Period 1 January 1961 - 1 April 1961

Unit Identification	Overtone of Operation	Electrode* Metal	Storage Temp. (°C)	Test Period (Days)	Series Resistance (ohms)	Total Aging (PP 10^{-7})	Remarks
E-5-3	5	Ag	60	14	36	-7.1	Poor unit, null unstable
E-5-4	5	Ag	60	19	22	-8.4	" " " "
E-5-5	5	Ag	60	84	22	-21.0	Unit leaking
E-5-7	5	Ag	60	84	17	-12.0	Poor unit, null unstable
E-5-8	5	Ag	60	21	50	-5.0	" " " "
F-5-1	5	Cu	60	60	39	+0.7	
F-5-3	5	Cu	60	60	40	-4.7	
F-5-4	5	Cu	60	60	20	+1.3	
G-5-1	5	Ag	60	7	30	-6.5	Bonding cement trouble
G-5-2	5	Ag	60	9	39	-0.1	" " "
G-5-3	5	Ag	60	7	20	-0.3	" " "
G-5-4	5	Ag	60	9	12	-0.6	" " "
G-5-5	5	Ag	60	9	17	-2.6	" " "
G-5-6	5	Ag	0	13	43	-2.1	" " "
G-5-7	5	Ag	0	13	46	-5.2	" " "
G-5-8	5	Ag	0	13	15	-2.8	" " "
G-5-9	5	Ag	0	13	39	-4.0	" " "
G-5-10	5	Ag	0	13	25	-2.4	" " "
H-5-1	5	Ag + Ag	0	7	41	-0.6	
H-5-2	5	Ag + Ag	0	7	53	-0.3	
J-5-2	5	Ag + Ag	60	7	17	-1.7	
J-5-4	5	Ag + Ag	60	7	39	+4.6	
J-5-5	5	Ag + Ag	60	7	14	-5.5	Leak at leads "patched" with glyptal
J-5-6	5	Ag + Ag	60	7	17	-1.8	
J-5-7	5	Ag + Ag	60	7	19	+2.2	
J-5-9	5	Ag + Ag	60	7	14	-1.1	Leak at leads "patched" with glyptal
J-5-10	5	Ag + Ag	0	7	40	-0.2	

* All metals deposited by evaporation.

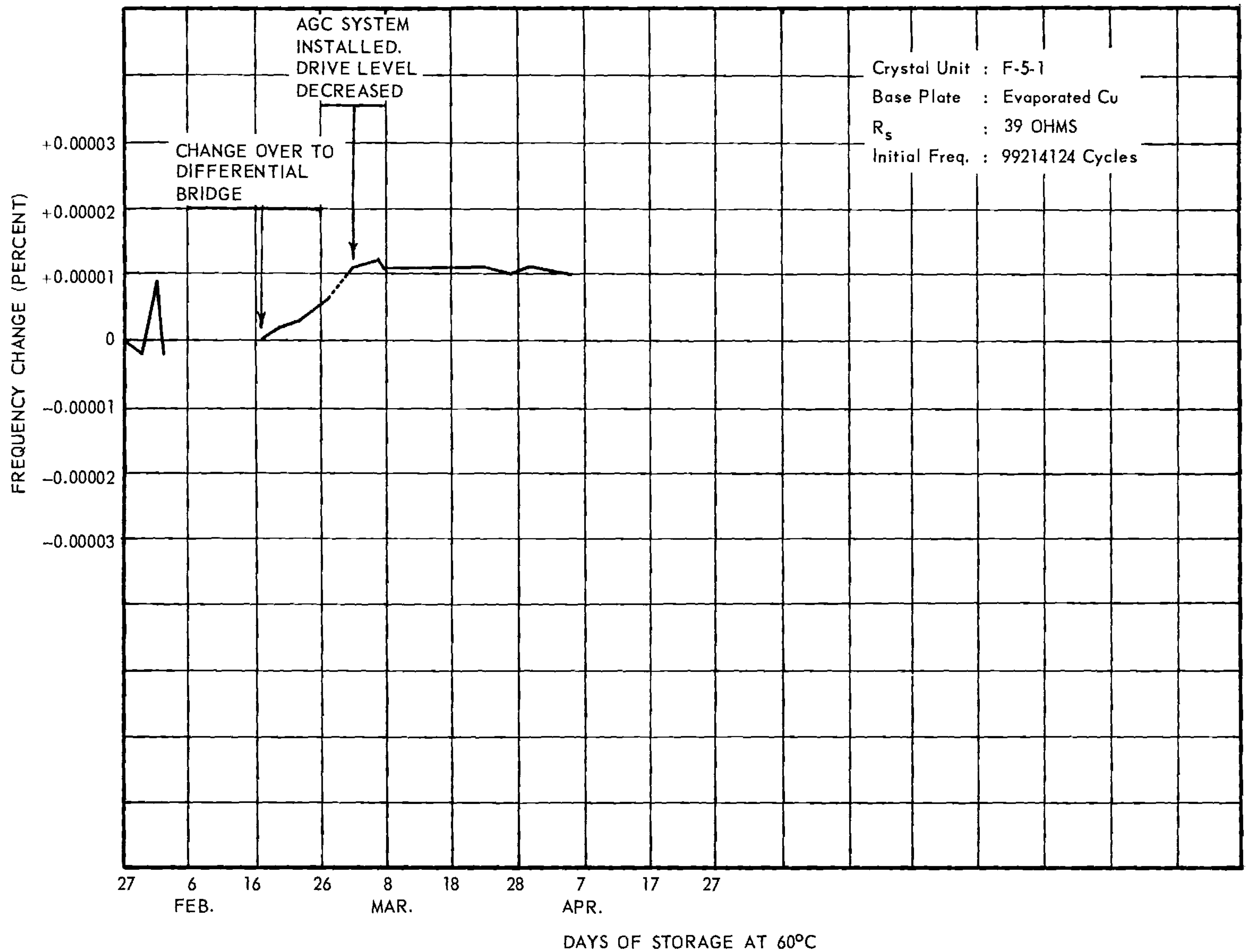


Figure 5. Aging data for resonator F-5-1-Cu, a copper plated fifth overtone unit operated at 60°C.

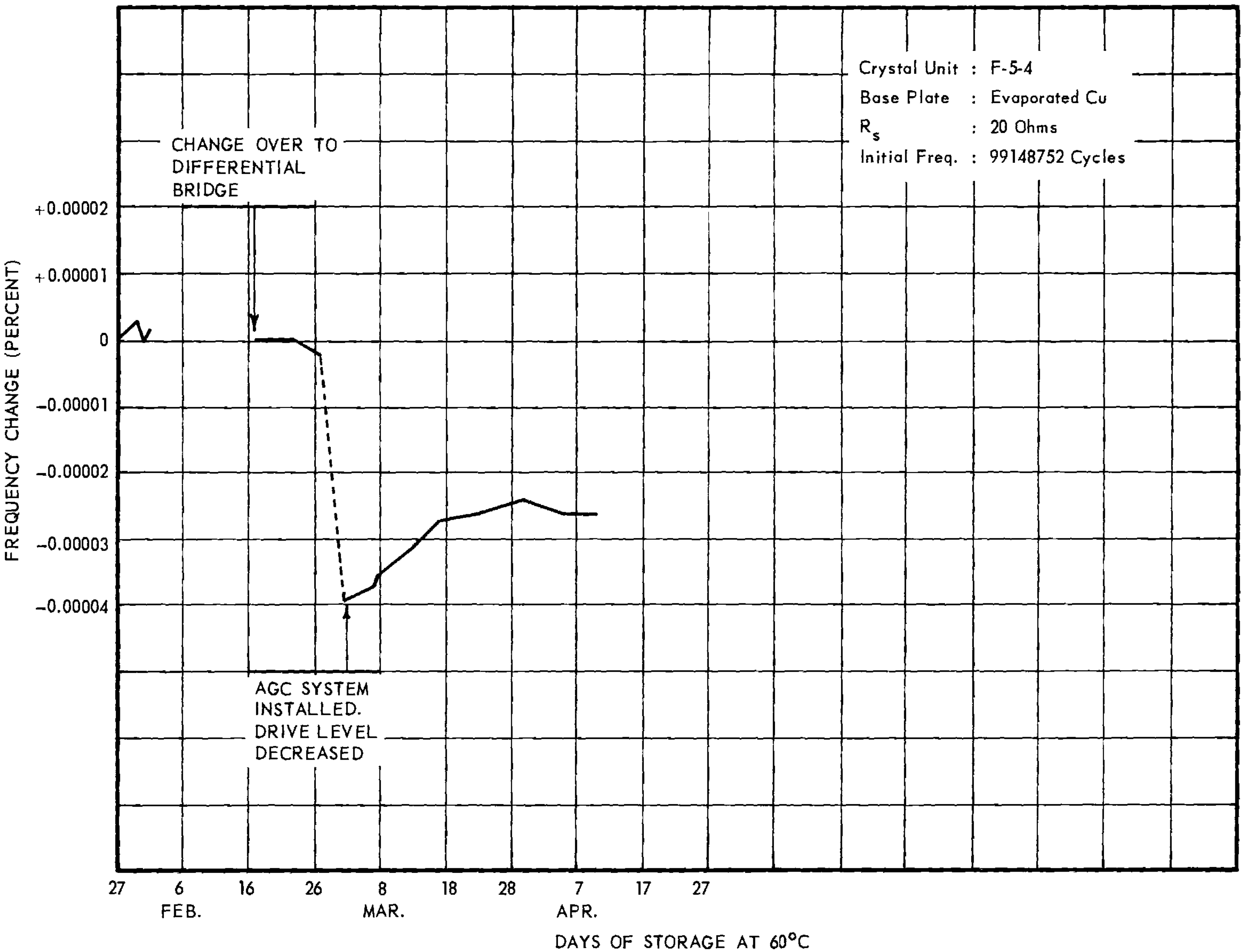


Figure 6. Aging data for resonator F-5-4-Cu, a copper plated fifth overtone unit operated at 60°C.

Data on resonators subjected to an environment incorporating temperature cycling through the range 0°C to 60°C daily will be reported in the fourth quarter.

5. Resonators Stored at 0°C

Aging data on some 15 resonators stored at 0°C have been obtained. A portion of these data are exhibited in Table I and a portion in Table II. All units included in these measurements were fifth overtone units. Aging of units of this mode has generally been relatively rapid at 60°C. However, at 0°C the frequency drift for seven units over a period of 32 days has been relatively low. Figure 7 displays the behavior of resonators A-5-9-Al. Essentially zero drift has occurred for this unit in 32 days.

In general, the drift of all units stored at 0°C has been small. R_s values as low as 10 ohms for 5th overtone units were noted for three of seven units.

When the 0°C oven was opened and the oven therefore cooled to the temperature of the surrounding freezer locker (about -15°C) a shift in frequency for particular units was observed. This is displayed in Figure 8 (C-5-5-Al). One hypothesis for this behavior is a condensation of vapor (probably water) on the unit at the very low temperature of the freezer. This hypothesis fits the data as displayed. The source of the vapor has not yet been defined; however, it is well known that water vapor may constitute a large part of residual gases in any vacuum system and the container sealing phase during fabrication may readily furnish vapor which is never fully exhausted through the small envelope exhaust tube employed.

6. Behavior of Resonators Exposed to Irradiation

a. Resonators exposed to radiation from 12,000 curie Cesium-137 source

The apparatus and procedures for irradiation of resonators was described in section B-3-a of this report. Nine of the more stable ninth overtone resonators, previously on aging measurement for periods of 210 to 240 days at 60°C, were selected for this experiment. Six of these units were plated with Al and three with Ag.

TABLE II

Aging of Old Resonators Stored and Measured at 0°C

Unit Identification	Fabrication Date	Test* Period (Days)	F** (PP 10^{-7})	Series Resistance (ohms)	Remarks
A-5-1	7-29-60	32	+0.0	~10	5th mode, Al Plating
A-5-3	7-29-60	32	-0.4	33	" " " "
A-5-9	7-29-60	32	-0.3	~10	" " " "
A-5-10	7-29-60	32	+0.0	19	" " " "
C-5-2	10-5-60	32	+1.1	39	" " " "
C-5-3	10-5-60	32	-0.8	30	" " " "
C-5-5	10-5-60	32	-1.8	~10	" " " "

* These units have been pre-aged at 60°C for about 120 days.

** Certain units stored at 0°C are quite sensitive to the temperature change when the oven is opened. The effect is to produce a "step" on the aging curve which may be either positive or negative. The temperature at the crystal sites drop well below 0°C when the oven is opened since the oven must be left in the freezer to prevent icing.

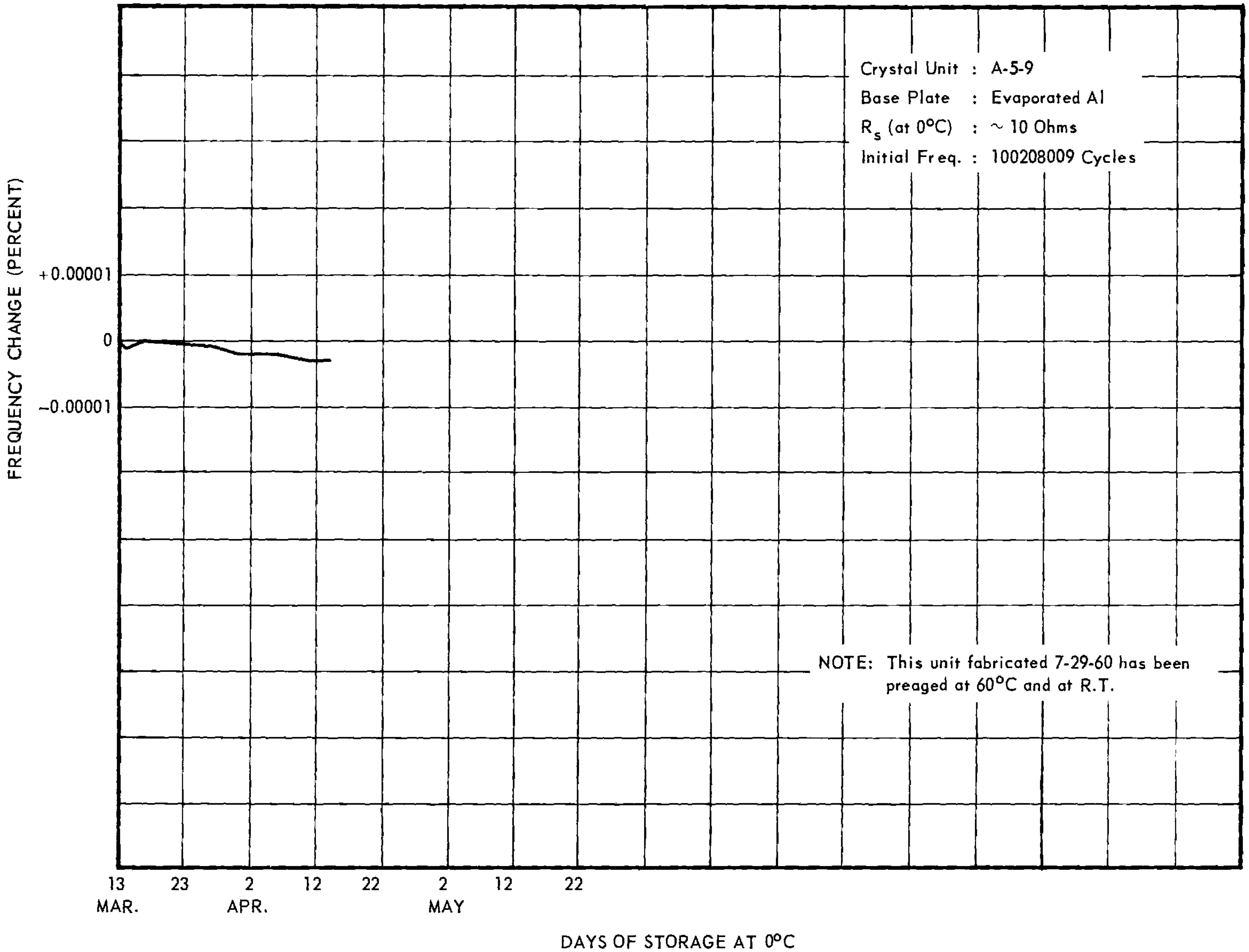


Figure 7. Aging data for resonator A-5-9-Al, an aluminum plated fifth overtone unit, stored at 0°C after previous aging (240 days) at 60°C.

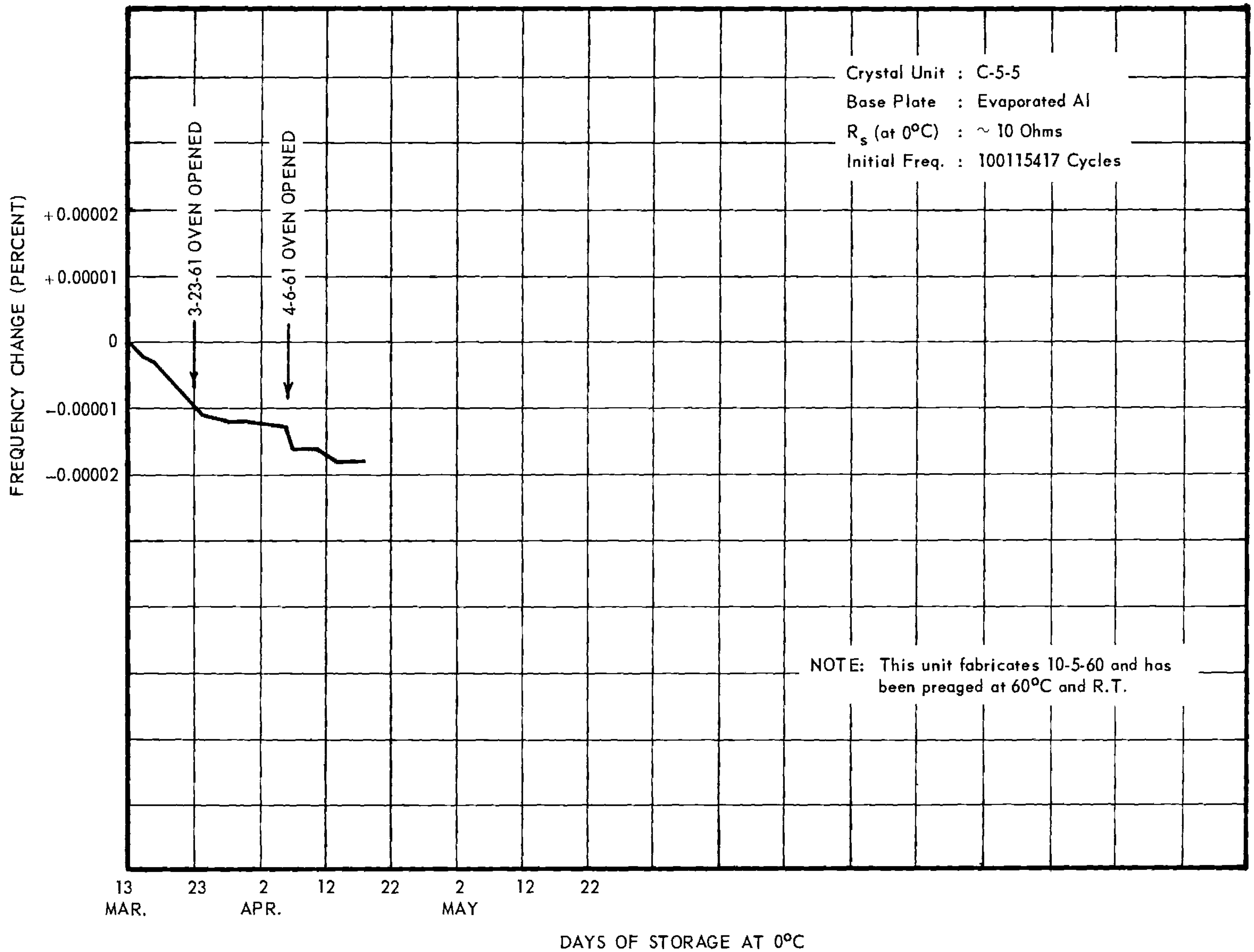


Figure 8. Aging data for resonator C-5-5-Al, an aluminum plated fifth overtone unit, stored at 0°C. Note peculiar drops in frequency observed when oven temperature drops below 0°C during period oven is open.

This group of units was divided into sub groups of three each and exposed to radiation from the Cesium-137 source at a dose rate of approximately 1.4×10^6 rad/hr. (Nearly the maximum of the source.)

The three sub groups were exposed for periods of 10 minutes, 20 minutes, and 30 minutes in the irradiation field.

The glass envelope of the units exposed for the longer time were smoky black. Those of the others were tinged with black or brown to a lesser degree.

The frequency shifts observed and the parameters of the units are exhibited in Table III. The correlation of shifts with time of exposure and the metal of the plating are shown in Figure 9.

It will be noted that shifts of 1000 cycles or 10 ppm were registered for the aluminum coated units and 700 cycles for the silver plated ones in 30 minutes. Although the data are scattered widely because of the relatively few specimens examined it is probable that a frequency versus time relation somewhat similar to that projected exists.

When three specimens were exposed at the much decreased intensity of 2.0×10^4 rad/hr for one hour, shifts of relatively small value, 1 to 30 cycles were noted. These shifts plotted on the same scale as Figure 9 are almost invisible. This particular intensity was selected as being equivalent to the intensity of beta bombardment in the Van Allen belt. This bombardment has the complexities suggested in the introductory paragraph preceding but gamma radiation is a major component of it since 600 Kev electrons are of sufficient energy to result in continuous spectrum gamma radiation from the satellite shell, whatever its constitution.

The resonators were replaced in the 60°C oven after exposure. Upward aging in general was observed. The total aging data on typical units is exhibited in Figures 10(A-9-9-Al) and 11(B-9-2-Ag).

In order to compare the behavior of these units with similar units exposed only to removal from the oven, storage at room temperature for the

TABLE III

Parameters of 100 Mc Quartz Resonators Exposed to Radiation of Various Type, Time and Dosage

Unit* Designation	Plating	Overtone	Source	Type Radiation	Exposure Time (Min)	Dosage rad/hr	ΔF** (Cycles)	ΔR_s ** (Ohms)	(PP 10^{-8})† 30 Days Aging Prior to Exposure	(PP 10^{-8}) Aging After Exposure	No. Days After Exp.	Remarks
A-9-2	Al	9	Cesium-137	γ	10	1.4×10^6	-545	+8	+0.0	+56	17	Glass discolored
A-9-3	Al	9	"	"	10	"	-340	+18	+0.0	+47	17	" "
A-9-4	Al	9	"	"	20	"	-527	+7	+8.0	+46	17	" "
A-9-6	Al	9	"	"	20	"	-323	+5	+7.0	+24	17	" "
A-9-7	Al	9	"	"	30	"	-1048	+16	-4.0	+62	17	" "
A-9-9	Al	9	"	"	30	"	-1097	+24	+3.0	+79	17	" "
B-9-2	Ag	9	"	"	10	"	-280	+10	-4.0	+30	14	" "
B-9-4	Ag	9	"	"	20	"	-730	increased	+0.0	+62	17	" "
B-9-6	Ag	9	"	"	30	"	-737	increased	+4.0	+25	17	" " Erratic after radiation
D-9-3	Ag	9	"	"	60	2.0×10^4	-29	N.C	±0.0	+5	14	No visible damage
D-9-4	Ag	9	"	"	60	"	-13	N.C	±0.0	+3	14	" " "
D-9-5	Ag	9	"	"	60	"	-18	N.C	±2.0	+5	14	" " "
F-5-3	Cu	5	Van de Graaf	Proton	~1/60	(1 Mev)	-32	N.C	-31.0	±0.0	7	" " "
F-5-4	Cu	5	"	"	~1/60	(1 Mev)	~1	-2.0	+7.0	+5	7	" " "

* All mounted in evacuated glass bulbs.

** Calculated from measurements at 60°C before and after exposure.

† Where aging has been ± 2 cycles or less, the aging rate is assumed to be essentially zero.

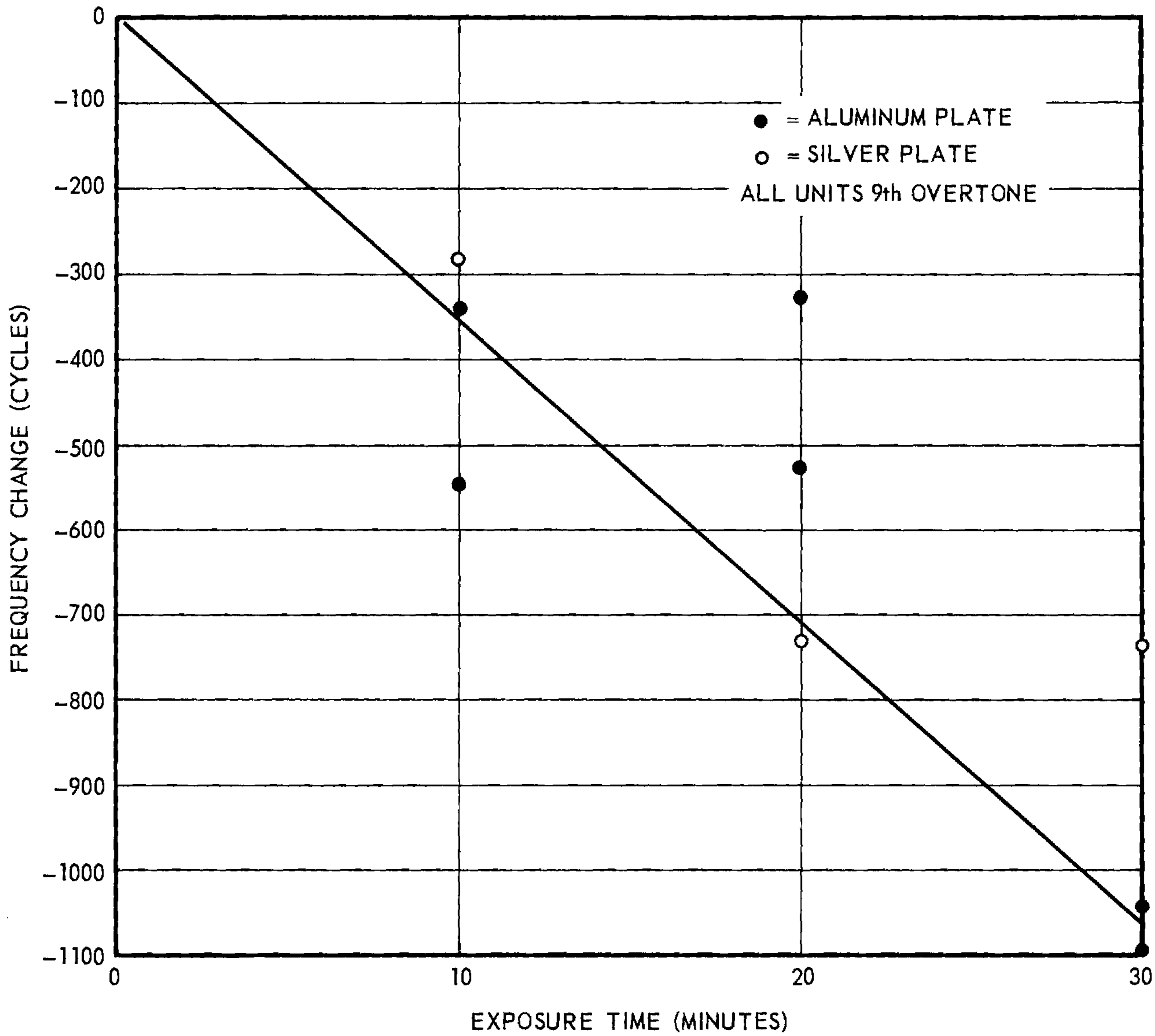


Figure 9. Frequency shifts occurring when selected resonators were exposed to radiation from the 12,000 curie Cesium-137 source.

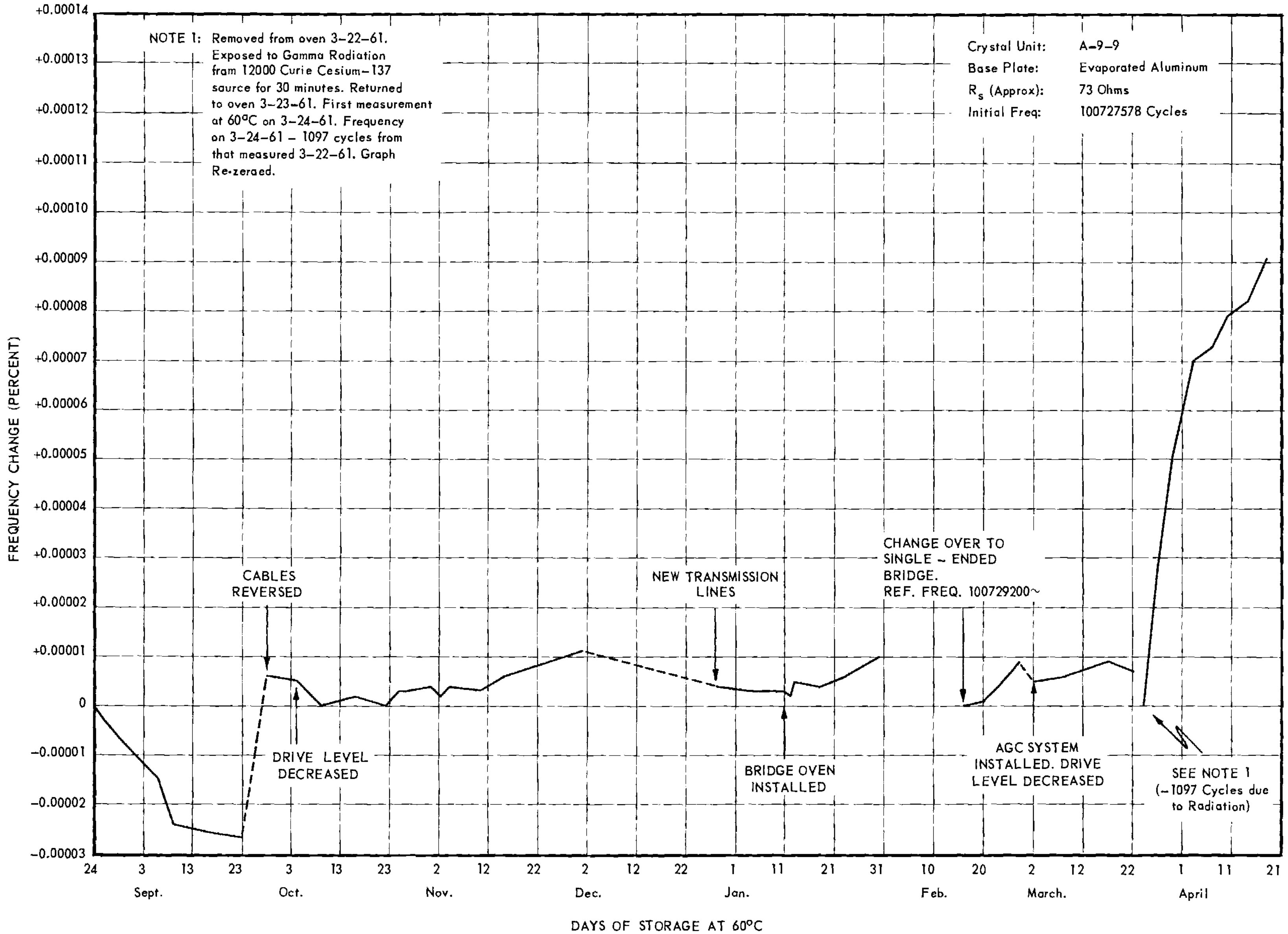


Figure 10. Aging of resonator A-9-9-A1, before and after exposure to radiation from Cesium-137 source (1.4×10^6 rad/hr for 30').

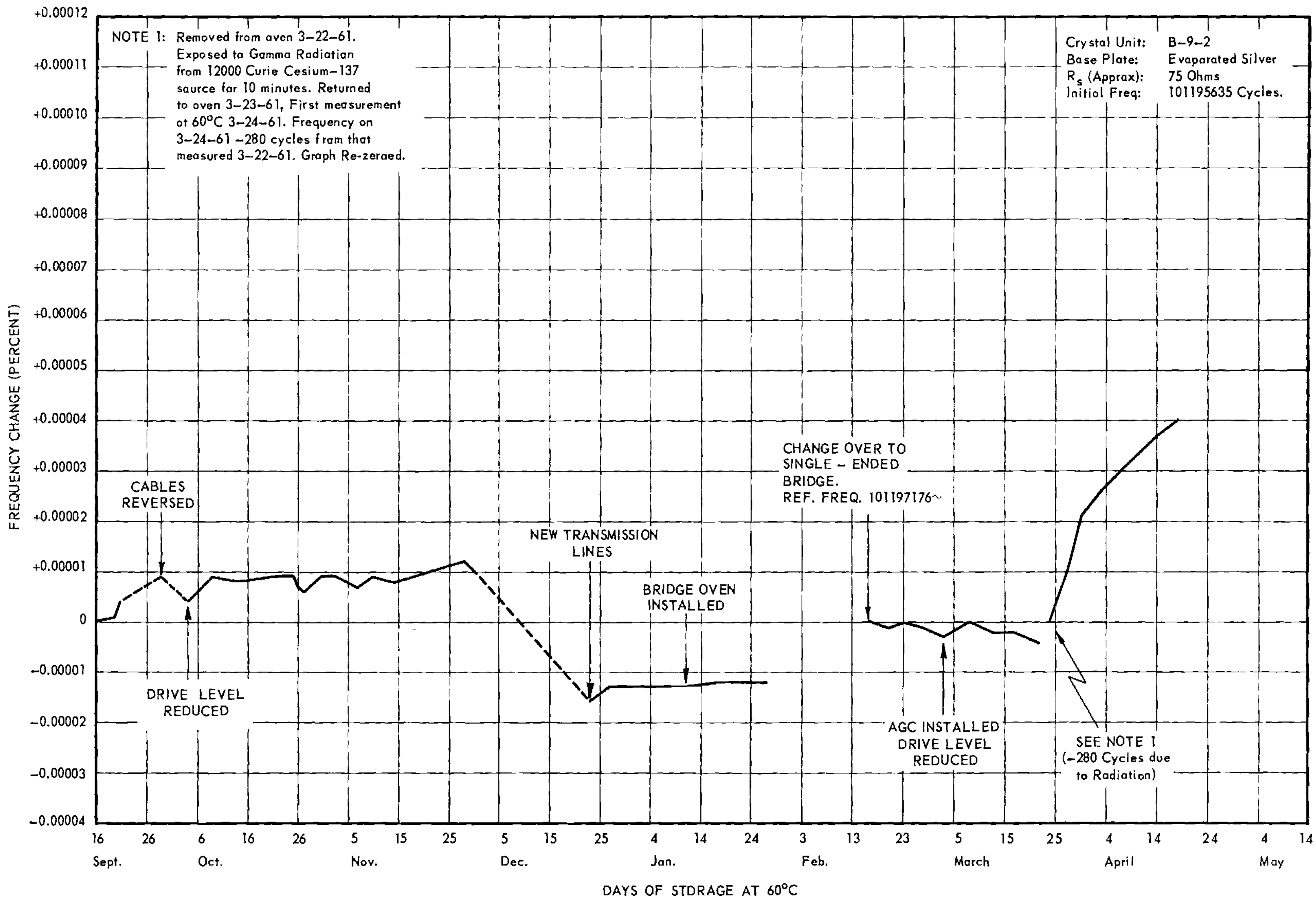


Figure 11. Aging of resonator B-9-2-Ag, before and after exposure to radiation from Cesium-137 source (1.4×10^6 rad/hr for 10').

same total period and return to the oven, resonator C-9-1-Ag was so treated. Its behavior is exhibited in Figure 12.

The shift of a resonator exposed at the lesser intensity of 2.0 x 10^4 rad/hr for one hour is shown in Figure 13 (D-9-5-Ag). The shifts of these units were quite small, averaging only 20 cycles for the three units.

b. Resonators exposed to 1 Mev proton beam

Two resonators were selected for this experiment. The magnetic field of the 1 Mev Van de Graaf Accelerator was adjusted so that the resonator could be bombarded by protons of 1 Mev energy at a current of 1 microampere. The dose received corresponds approximately to 100 times that received from the 40 Mev protons but is comparable to the total dose received in one hour in the Van Allen Belt. This equivalent exposure was obtained in the experimental set-up in one second. However, timing control for such short intervals was not good and the actual time of exposure was one or two seconds.

For these two resonators shifts of -1 and -33 cycles were observed. These are very small relatively speaking. Additional measurements will be made during the fourth quarter.

d. Other Studies

1. Bonding cement problems and experiments

For some time all crystal units fabricated at Georgia Tech were bonded with Hanovia No. 2* cement. This cement is a silver dispersion in distilled water and is recommended for use when the vapors from organic thinners may present a problem. Until recently, results with respect to aging and reliability have been very good. However, a supply of cement received 18 October 1960 and first used on C-9 group fabricated 14 November 1960 has given consistently poor results. In some cases crystals which would operate unbonded failed to operate after bonding. One group of silver-plated, 5th mode crystals (G-5) bonded with Hanovia No. 2 cement

* Englehard Industries, Inc., East Newark, N. J.

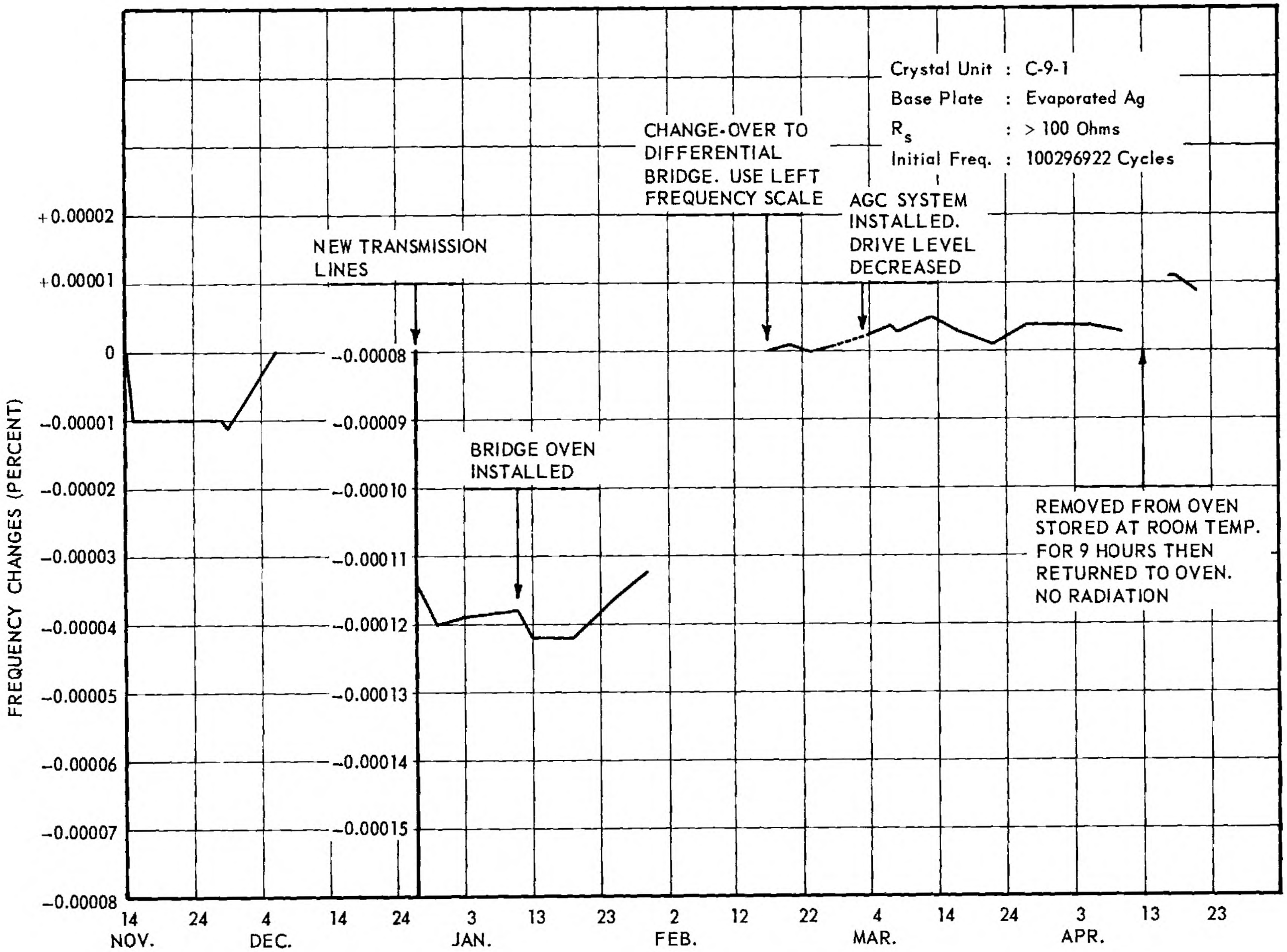


Figure 12. Aging of resonator C-9-1-Ag, before and after removal from oven and storage at room temperature without irradiation.

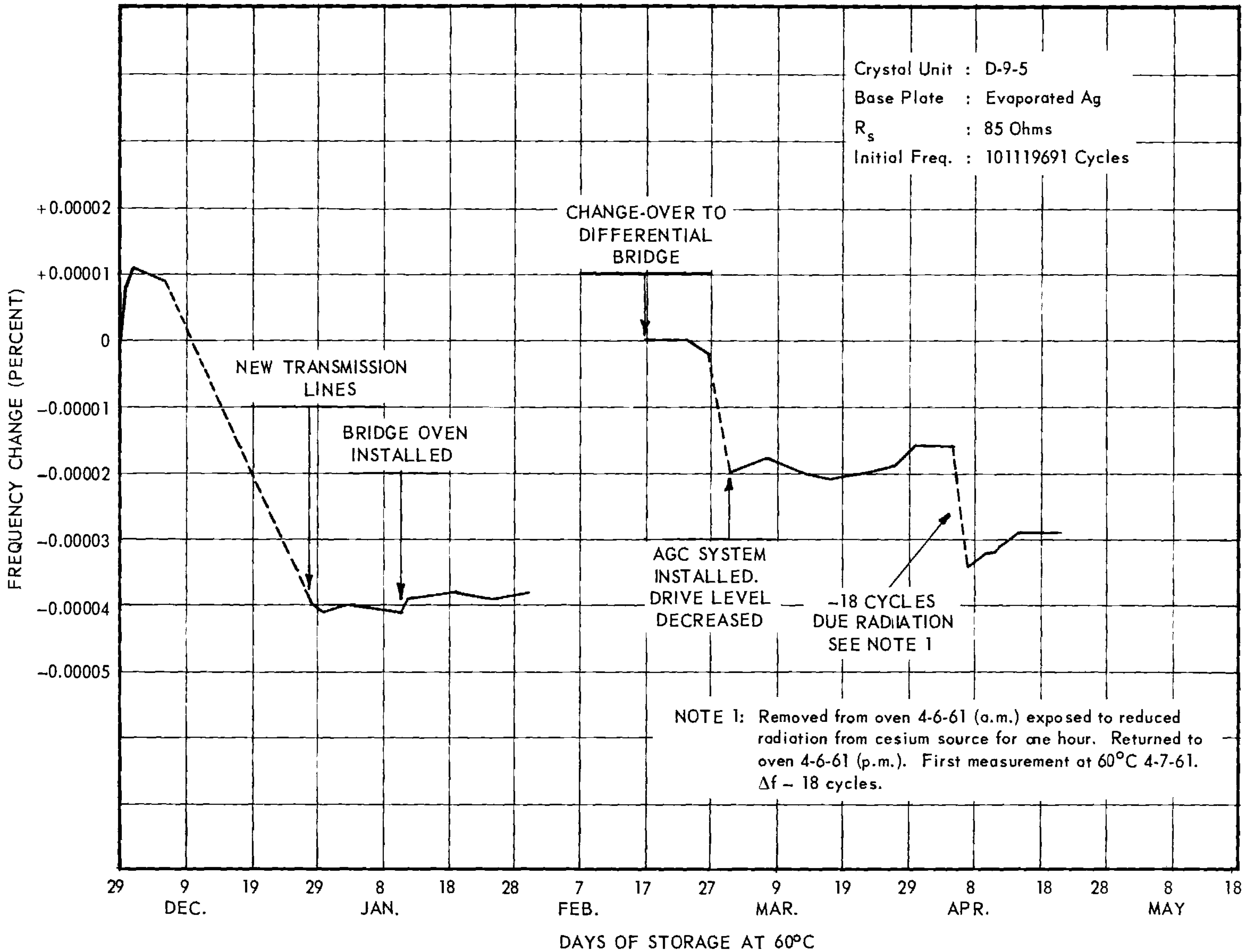


Figure 13. Aging of resonator D-9-5-Ag, before and after exposure to radiation from Cesium-137 source at lower intensity (2.0×10^4 rad/hr for one hour).

were completed with 100% initial yield. Aging measurements especially those at 0°C indicated some basic trouble. (See Table L) All G-5 units were removed from the aging ovens for inspection. Observation with a stero-microscope revealed that the plating near the cement and extending in some cases into the spot had "buckled". This action is probably due to one or both of the following causes:

a. shrinkage of the cement while drying;

b. diffusion of some component in the cement into the silver plating.

The behavior observed is illustrated in Figures 14A and B. In view of the fact that the units stored at 0°C aged more than those at 60°C some weight is given to the first hypothesis.

The manufacturer of Hanovia No. 2 cement has been informed of the problem and invited to comment.

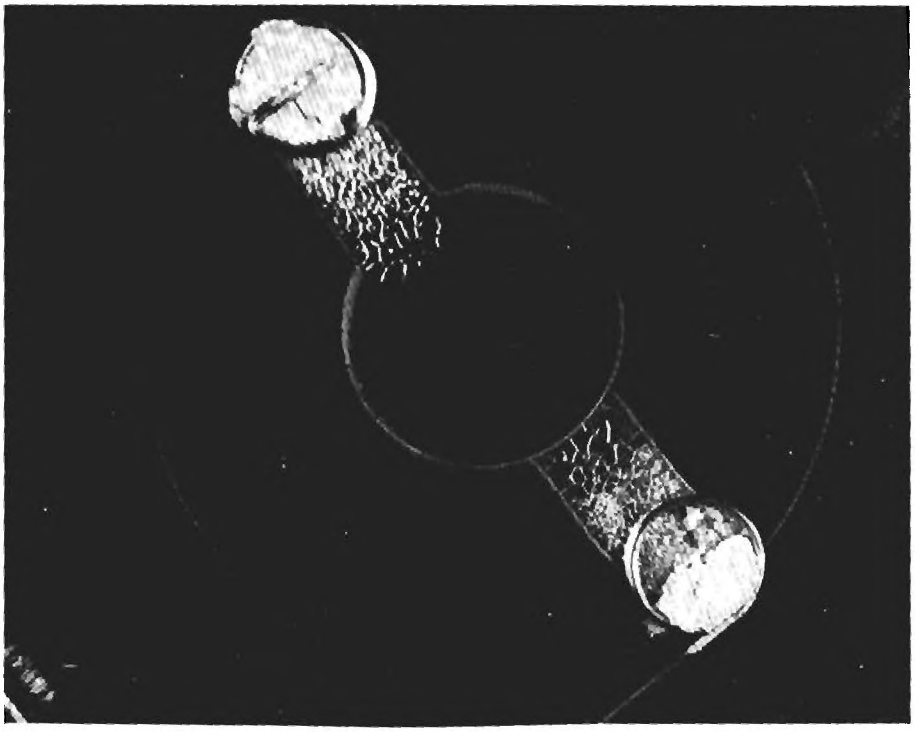
A change of bonding cement was certainly indicated. Fortunately a supply of du Pont No. 5504 a silver-loaded, epoxy cement was on hand. Preparations for its use were made by bonding several pieces of 28 gauge nichrome wire to clean glass plates and then curing the cement for three hours at 300°F (150°C). Qualitative measurements indicated that the cement made a bond having excellent electrical and mechanical properties. One of the plates was returned to the oven and the cement heated three hours at 600°F. The condition of the cement was still very good.

One group of units bonded with No. 5504 A cement has been completed and aging information will be available for the progress letter for April 1961.

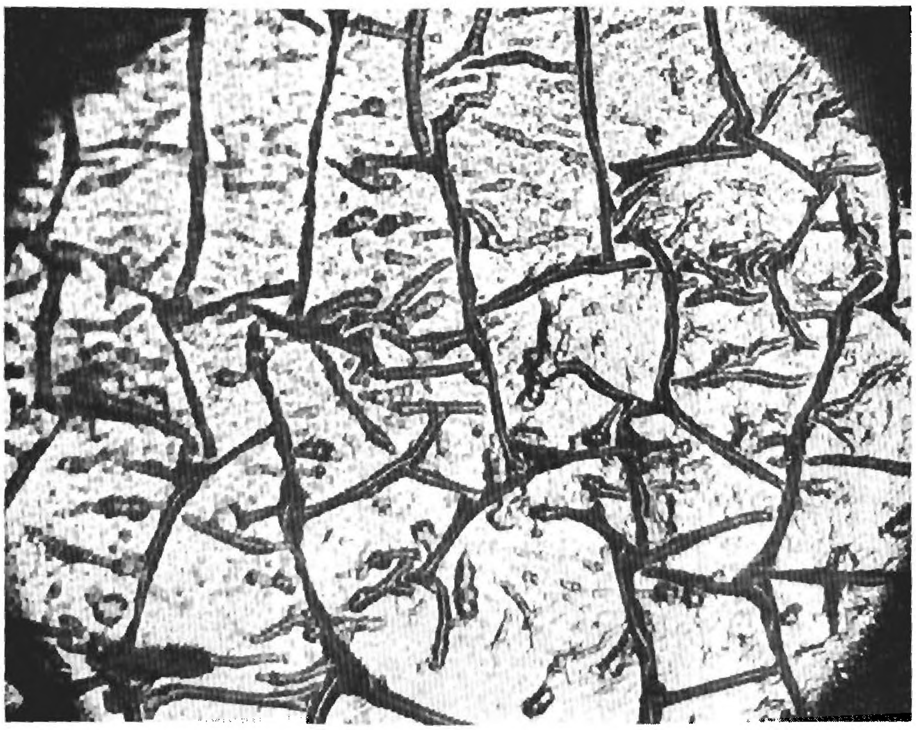
2. Problems Encountered with the New Glass Stems*

The new stems were first used with crystal group D-5 fabricated 10 October 1960. Some problems were initially encountered making good seals to the bulb. A method of making the seal was developed that now produces an acceptable yield of 80% or more. When the bulb sealing

*Obtained from the WRW Scientific Glass Co., Inc., 10 Sagamore Hill Drive, Port Washington, N. Y. (Bliley Drawing No. K-475-M)



A. General view



B. Enlarged view of damaged area (150x)

Figure 14. Micrograph of quartz resonator exhibiting damage to bonding cement.

problem was virtually eliminated, a basic flaw in the stems as received was found. Studies have indicated that 20% and possibly more of the stems leak at the glass to metal lead seals. Such leaks are usually quite small and are usually easily sealed with glyptal. Actually, glyptal is applied to all seals after the units are leak tested individually before vacuum baking. An epoxy resin is presently being considered to replace the glyptal. However, when attempting to fabricate highly stable VHF crystal units one should not be forced to contend with leaking glass holders.

The stems previously used* and the companion bottles were very easily sealed and no lead trouble was encountered. Procurement of a new stock from the original supplier is now underway. A change back to the old stems will be made at the first opportunity.

*Obtained from General Electric Lamp Division, Nela Park, Cleveland, Ohio.

V. CONCLUSIONS

Adoption of the single-ended capacitance bridge and the application of automatic gain control to the driving oscillator in the frequency measurement apparatus have resolved the frequency measuring problem. Measurements of ± 2 parts in 10^8 are repeatable.

The yield of high stability 5th mode 100 Mc resonators during the quarter has remained poor, partly due to a batch of poorly bonding cement and leaks in glass stems. The initial yield has now been improved to approximately 80 percent or higher. A supply of new bonding cement and new glass envelopes and stems of higher quality is expected to improve the yield to a higher and more consistent value.

Selected resonators, aged at 60°C, have continued to show minimal aging of less than 1 part in 10^7 in six months; and very low aging has been experienced at 0°C for similarly fabricated units.

Resonators exposed to intense radiation from a Cesium-137 source (1.4×10^6 rad/hr) shift downward in frequency in accordance with time of exposure. Aluminum plated units shift more than silver plated ones. Shifts of 10 ppm were observed after 30 minutes exposure at this intensity.

At a lower intensity (2.0×10^4 rad/hr), approximately equivalent to that of the Van Allen belt, shifts of only an average of about 20 cycles were experienced after an exposure of 1 hour.

Resonators exposed to a 1 Mev proton beam of 1 microampere for one second shifted downward only 1 to 33 cycles. This is estimated to be equivalent to about one hour's exposure in the Van Allen belt although not equivalent in particle energy (60 Mev).

After exposure at the maximum intensities resonators drifted upward at rates of about 2 to 10 pp 10^8 per day. These undergoing smaller negative shifts initially because of the lower intensity of radiation exhibited much smaller recovery rates.

VI. PROGRAM FOR THE NEXT INTERVAL

The plan for the succeeding quarter is to complete more comprehensive studies of the effects of radiation and to perform the temperature cycling studies.

Effort to improve fabrication quality and yield will be continued. Predominant emphasis will be placed on 7th mode resonators.

A paper will be prepared and presented at the Fifteenth Annual Frequency Control Symposium.

VII. IDENTIFICATION OF KEY TECHNICAL PERSONNEL

The following persons have been employed on this project during its third quarter for the times indicated.

Name	Position	(Hours)
Richard B. Belser	Project Director	170
W. Bruce Warren	Research Engineer	135
Walter H. Hicklin	Asst. Research Engineer	428
James O. Darnell	Research Assistant	512
Carol M. Shirley	Technician	420
W. Donald Dawson	Student Assistant	74

Mr. Belser has been associated with resonator aging studies sponsored by USASRADL for over ten years and has been assisted by Mr. Hicklin for approximately nine years. Mr. Warren, an electrical engineering graduate of Georgia Tech with an M. S. degree, has served in the capacity of project director on projects sponsored by both Signal Corps and Air Force Agencies for a number of years and has had recent industrial experience with a leading communication manufacturer.

Respectfully submitted,

Richard B. Belser
Project Director

Approved:

Vernon Crawford
Head, Physics Branch
Physical Sciences Division

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Erie, Pennsylvania

1 Bell Telephone Laboratories
Whippany, New Jersey
Attention: Dr. J. C. King

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ARPA Order No. 54-60
Engineering Experiment Station
Georgia Institute of Technology
10 June 1960 to 30 June 1961

Contract No. DA-36-039-sc-85363
(Unclassified)

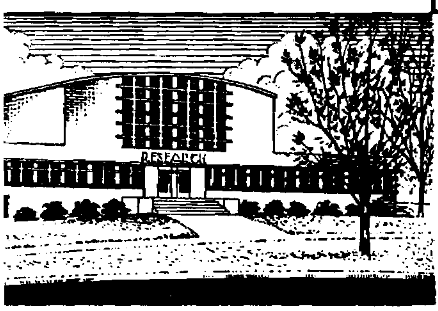
Stability Studies of Quartz Crystals for Satellites

By
R. B. Belser and W. H. Hicklin

Final Report
Contract No. DA-36-039-sc-85363
10 June 1960 to 30 June 1961
Georgia Tech Project No. A-508

Placed by the U. S. Army
Signal Research and Development Laboratories
Under ARPA Order No. 54-60

"The work performed under this contract was made possible by the support of the Advanced Research Projects Agency under Order No. 54-60 through the U. S. Army Research and Development Laboratory."



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I. PURPOSE

The purpose of this project is to develop AT-cut quartz resonators of 100 Mc frequency with stabilities suitable for operation in satellites or other space vehicles. Initial target stabilities are a maximum frequency deviation of ± 0.3 parts per million per year and ultimate desired stabilities of ± 0.1 parts per million per year. These stabilities are to be maintained while the resonator is subjected to temperature cycles of 0° to 60°C one or more times per day and exposed to radiation similar to that of the Van Allen Belt.

Resonators operated in the 5th, 7th, and 9th overtone modes will be investigated. Plating metals are to be silver or aluminum. Effects of radiation similar to that in the Van Allen Belt on the stability of the 100-Mc resonators will be studied.

II. ABSTRACT

Over one hundred 100-Mc AT-cut quartz resonators have been fabricated and stored in constant temperature ovens at 0°C and 60°C or cycled through the temperature range 0°C to 60°C once daily. These units have consisted of resonators operated at the fifth, seventh, and ninth overtones and were plated with silver, aluminum or copper.

Initially studies were made of units coated only with a base plating, but subsequent units were overcoated to frequency with the metal of the base plating or, for some of the aluminum plated resonators, with silver or gold.

Frequency measurements have been made by a bridge and counter method derived from that of Robertson* utilizing the Crystal Impedance Meter TS-15 as the oscillator. The measuring apperatus was subsequently changed to incorporate a single ended bridge and automatic gain control for the oscillator. The standard frequency was derived from a bank of three Western Electric O-76 U oscillators monitored against the 18-kc signal of radio station NBA, Balboa, Canal Zone; an Atomichron served as a standby standard. The NBA propagated signal was found to be received in Atlanta, Georgia, to an accuracy of 6 parts in 10^{10} by comparison with the Atomichron.

The more stable units have been ninth overtone units coated with silver. Several of these have held ±5 parts in 10^{8} for a period of more than 180 days.

Initial studies on radiation damage were conducted by exposure of 12 stable ninth-mode units to the radiation from a 12,000-Curie Cesium-137 Research Irradiator (approximately 1×10^{6} rad/hr.). A dosage for 30 minutes

*Douglas W. Robertson, Final Report, U. S. Army Signal Research and Development Laboratories Contract No. DA-36-039-sc-56730, Georgia Institute of Technology, 1960.

at the highest intensity available resulted in a negative frequency shift of 5 to 10 ppm. Aging at 60°C thereafter was upward at a rate of about $+2 \times 10^{-8}$ per day. For a dosage of one hour at a lower intensity, more in agreement with that to be expected in the Van Allen Radiation Belt (2×10^{4} rad/hr.), shifts of about -2×10^{-7} were experienced. Recovery was at a rate of about $+1 \times 10^{-8}$ per day initially. The aluminum plated units were more adversely affected than the silver plated ones. Subsequent irradiation at 1.6×10^{6} rad/hr for periods of 15 to 24 hours induced first negative and then positive frequency shifts indicating that a saturation condition was reached. No post-saturation aging measurements were completed, however.

Measurements of frequency changes induced by 1 Mev proton bombardment (1 μa) for periods up to four minutes generally resulted in small positive shifts (a few parts in 10^{7}) in frequency.

III. PUBLICATIONS, LECTURES, REPORTS AND CONFERENCES

A paper entitled "Stability Studies of Quartz Crystals for Satellites" was presented at the 15th Annual Symposium on Frequency Control held at Atlantic City, New Jersey, May 31 - June 2, 1961, by Mr. R. B. Belser and Mr. W. H. Hicklin. This will be published in the Proceedings of the Symposium.

Mr. Belser, project director, visited the U. S. Army Signal Research and Development Laboratories at Fort Monmouth, N. J., for a conference on the project with Dr. G. K. Gutwein, Mr. M. Bernstein, Mr. J. M. Stanley and Mr. P. E. Mulvihill on 1 July 1960. The program and facilities available for the project were discussed. It was agreed that work would proceed essentially as outlined in the proposal. Mr. Belser presented a summary of aging data and explained the correlation between leak measurements obtained by the vacuum oil leak test and the stabilities obtained for 600 industrially procured 16.5 Mc resonators.

Subsequently on 31 August 1960 a second conference was held at the same place between the same parties with the exception of Mr. Stanley. Progress to date and procurement sources for better 100-Mc quartz blanks were discussed. Plans for the continuation of a parallel aging study of 16.0 Mc resonators at the fundamental frequency and at the third and fifth overtone modes were outlined.

Mr. P. E. Mulvihill, Project Engineer, visited the Georgia Institute of Technology on 6 March 1961. The progress and work of the project were examined and discussed. The radiation facilities were visited. Tentative plans for increased oven capacity for aging studies of commercially fabricated units at 85°C were described and considered. No action was taken on this matter pending further instructions.

IV. FACTUAL DATA

A. Introduction

In conjunction with the satellite program a need exists for quartz resonators of 100 Mc frequency exhibiting aging changes of less than 3 parts in 10^7 per year; a still more exacting requirement envisions units aging less than 1 part in 10^7 per year. In addition, delineation of expected aging that might be encountered due to temperature cycling daily over the range 0°C to 60° C and because of radiation damage from the Van Allen Belt was desired.

Previous experience with 16.5 Mc resonators had shown that resonators stable to within ±5 parts in 10^7 per year could be fabricated by techniques developed at the Georgia Institute of Technology under U. S. Army Signal Corps Contracts Nos. DA-36-039-sc-64613, DA-36-039-sc-74946, and DA-36-039-sc-78905. Fabrication of resonators by similar but improved methods was expected to solve the problem.

B. Apparatus and Procedures

1. Plating and Storage Equipment

The apparatus consisted of two vacuum systems for plating the resonators, ovens for storage of resonators at 0°C, at 60°C, and for cycling over the temperature range 0°C to 60°C one complete cycle daily, the frequency standards and measuring equipment, and facilities for irradiating resonators with gamma rays and bombarding them with electrons or protons.

The base plating vacuum chamber as shown in Figure 1 was a four-inch cross of pyrex pipe in which the crystals to be plated were suspended centrally by a shaft and mask assembly. Heater and evaporation filaments on each side

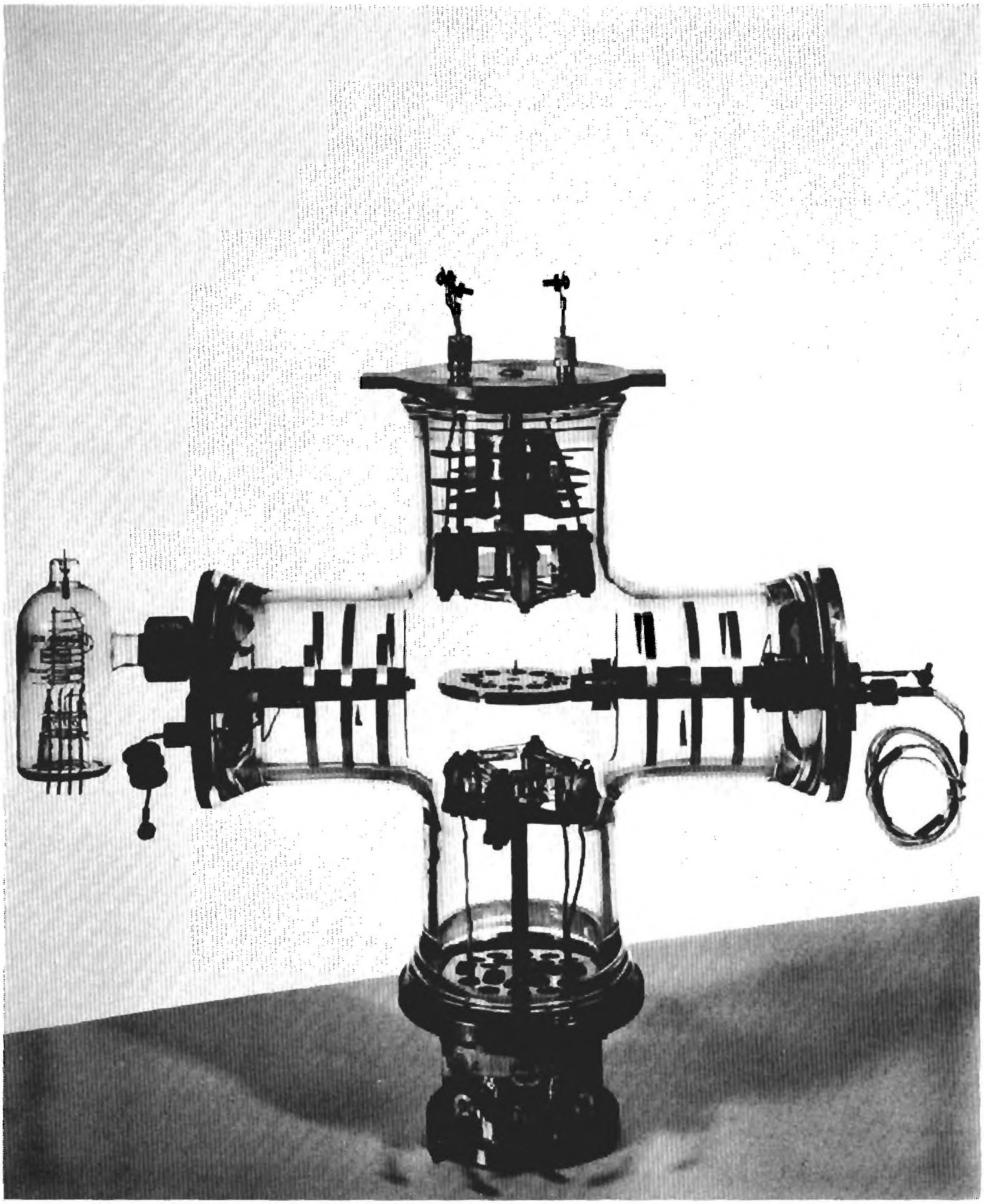

Figure 1. Vacuum chamber for base plating quartz resonators.

of the mask were provided respectively for radiant heating of the blanks to a selected temperature (monitored by chromel alumel thermocouple in the plane of the mask) and for subsequent plating. The chamber was fitted, in addition, with a liquid nitrogen cold trap and with filaments for evaporating getter substances.

The vacuum gauge tube with its entrance immediately to the chamber registered in the low 10^{-7} mm of mercury pressure region when the trapping and gettering actions were used in addition to the normal pumps. Plating was carried out in the lower pressure ranges. The system was evacuated initially by a four-inch oil diffusion pump and a forepump.

The 0° and 60° constant temperature storage ovens were constructed of five nested aluminum boxes insulated with 1/4" felt. Heaters of nichrome wire were wound around the second and fourth boxes and insulated therefrom with fiber glass tape*. Temperature control of the heater circuit was by means of a mercury thermostat accurate to approximately ±0.01°C.

Coaxial leads were passed through the successive bases of the nesting boxes for connection to 36 ceramic two-pin sockets to be used as resonator positions. The successive tops were arranged for rapid removal and replacement. The 0°C oven was located in a freezer compartment where the ambient temperature was approximately -17°C.

The cycling oven, similarly located, was constructed of only two lightly insulated nesting boxes since it was necessary for it to change temperature over the range 0°C to 60°C fairly rapidly. A motor driven thermostat in the oven power supply made it possible to control the rate of temperature increase and decrease at a rate of approximately 5°C per hour completing one cycle each

*Scotch Brand Thermosetting No. 27.

24 hours. However, the rate could be adjusted for other periods if desired.

2. Frequency Measuring Equipment

The frequency measurement equipment was derived from the bridge system previously used for measurement of 16.5-Mc resonators to approximately 2 parts in 10^7. The standard frequency was derived from three Western Electric oscillators, Type 0-76-U, monitored against Radio Station NBA, Canal Zone, and checked periodically with a National Atomichron. The adaptation of the original bridge system which utilized coaxial half-wave connector lines to the resonators in the oven sites revealed a temperature sensitivity in both the lines and bridge assembly. The bridge was redesigned to the single ended configuration shown in Figure 2. The crystal loop, not incorporating the resonant lines, is relatively insensitive to variations in the lines due to temperature, humidity, and time. The frequency of the crystal is therefore little affected by such changes.

A second improvement has been the adoption of an automatic gain control circuit for the oscillator screen voltage, shown in Figure 3.

This feature decreased the required crystal drive level and the time needed for measurements. Frequency measurements are considered accurate to ± 2 parts in 10^8.

3. Irradiation Facilities

The radiation sources available for this work were a 12,000-Curie Cesium-137 Research Irradiator and a 1 Mev Van de Graaff Accelerator.

The Cesium-137 Research Irradiator* consists of twelve 5/8-inch diameter brass tubes surrounding a 1-7/8-inch brass tube. Around the 5/8-inch tubes

*R. C. Palmer and R. W. Carter, International Journal of Applied Radiation and Isotopes 9, 123 (1961).

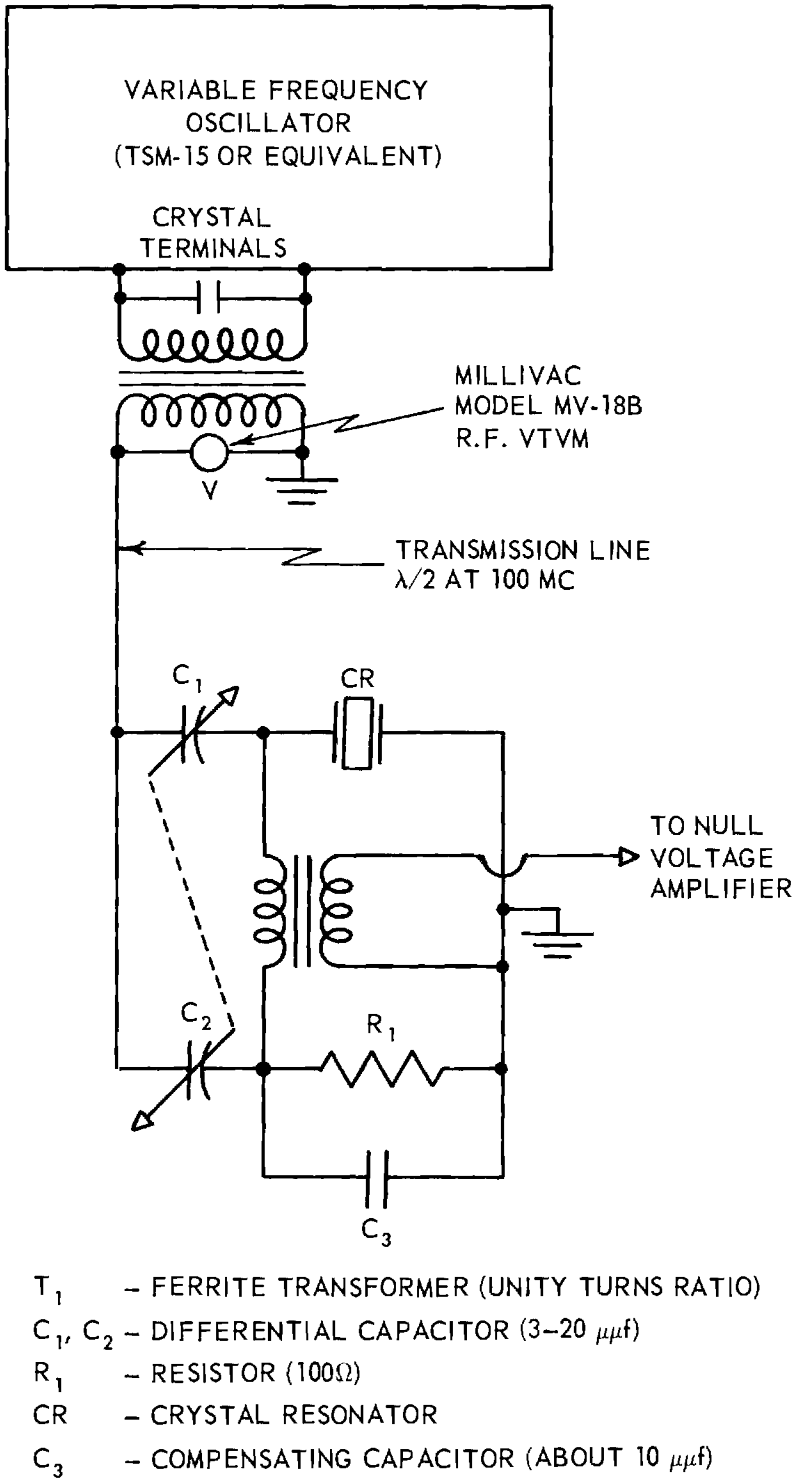

Figure 2. Single-ended bridge for improved measurement accuracy at high frequencies.

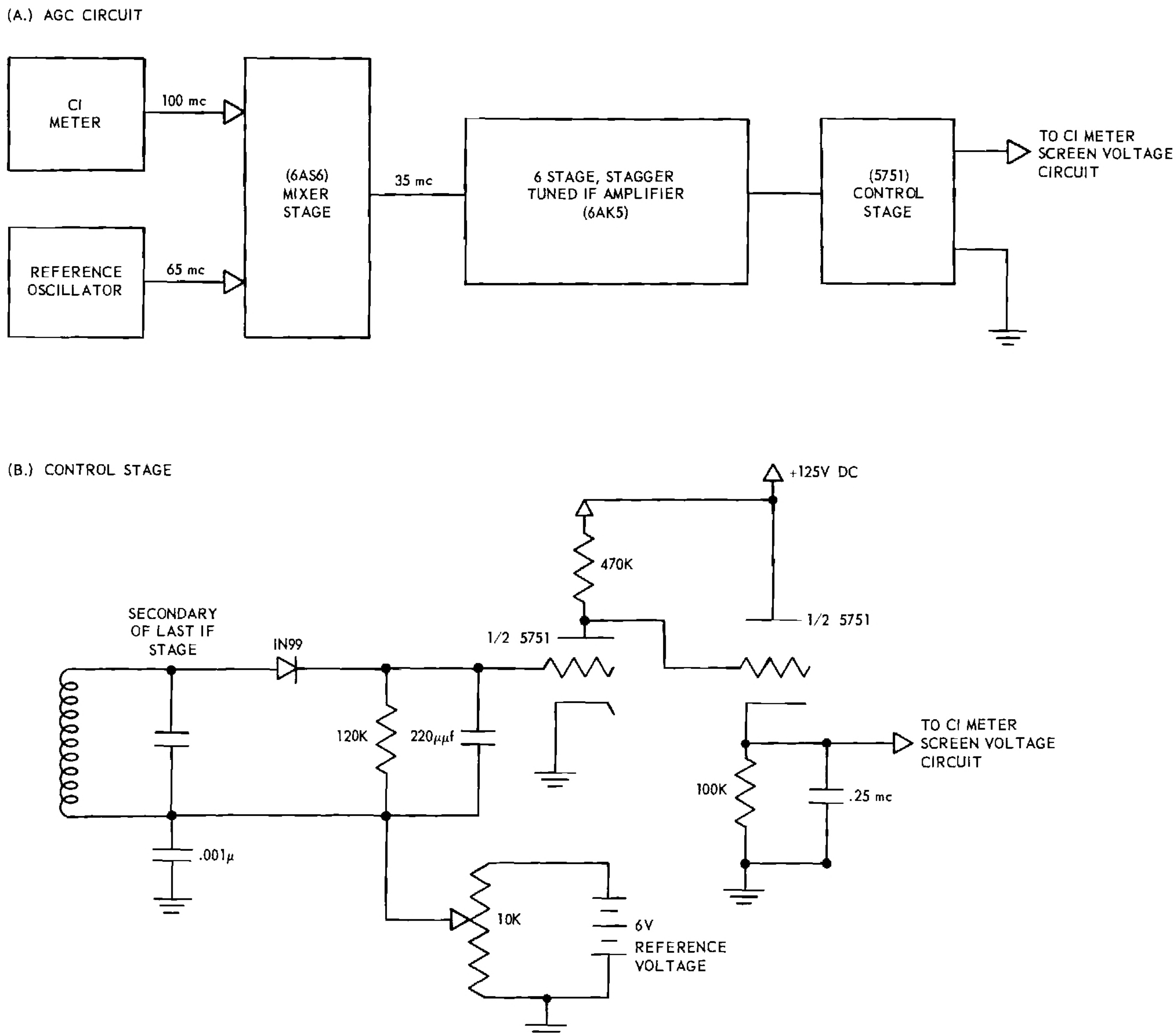

Figure 3. Block diagram of the AGC circuit used on the bridge driving oscillator and the circuit of the control stage.

are twelve additional 1-inch diameter brass tubes. This tube bundle extends 8.5 feet into the ground. Twelve Cesium-137 sources (approximately 1000 curies* each) are contained in the 5/8-inch diameter tubes at a depth of 8 feet below floor level. Samples to be irradiated are lowered into either the center tube or one of twelve outside tubes. Figure 4 shows the dose variance with depth in a well of the irradiator. By varying the placement of the sample any dose rate from 1.4×10^6 rad/hr to 10^{-3} rad/hr (not shown) can be obtained. This range is such that any "total dose rate" of the Van Allen Belt can be achieved.

The Van de Graaff Accelerator, which is housed in a specially designed facility in the Radioisotopes Laboratory, is a 1-Mev positive-ion accelerator. It contains a positive-ion source that can provide a continuous ion current of up to 70 microamperes. With hydrogen as the source gas, this current is about two-thirds protons (H^+ ions) and one-third molecular ions (H_2^+). Currently in use is a mixture of ordinary and heavy hydrogen (deuterium) so that the beam normally contains about 25 microamperes each of protons and deuterons, together with lesser amounts of the molecular ions H_2^+, HD^+, and D_2^+. In the experiments reported a current of only about 1 microampere was used.

The beam emerges vertically from the bottom of the machine and passes through an evacuated tube into the target room below, where it is deflected 90° into a horizontal path by a large electromagnet. By varying the magnetic field, the beam directed to the exit portal may be chosen at will to include either the mass-1 beam of protons or the mass-2 beam of deuterons (and H_2^+ ions). The magnet can be rotated about a vertical axis, so that the emerging beam

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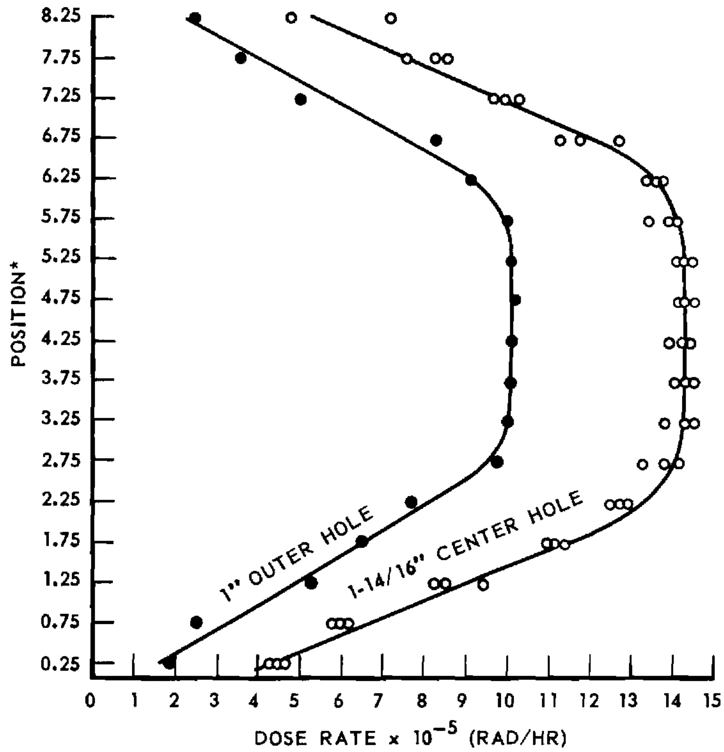
*One curie equals 3.7×10^{10} disintegrations per second, the rate of disintegration of 1 gram of pure radium.



* The position is measured upward in inches from the bottom of the sample space within the sample carriers.

Figure 4. Dose variance with vertical placement in the well of the Cesium-137 gamma irradiator.

can be directed to any azimuth, and it may be directed through any of several ports in the target-room wall into the adjacent physics laboratory.

C. Experimental Work

1. Resonator Fabrication

a. Base Plating

Crystal plates were cleaned with hot chromic acid, thoroughly rinsed in boiling distilled water, rinsed with methyl alcohol and dried with hot air. Detergents were not used because of the difficulty of completely rinsing the solution from the quartz. Loading into the plating mask and chamber was completed as quickly as possible.

Base plating was conducted in the following sequence of operations:

(1) The primary trap was loaded with dry ice and acetone.

(2) Evacuate chamber to a pressure of 10^{-5} mm of mercury.

(3) Flush system at least twice with dry argon and reevacuate.

(4) Set substrate heaters on auto-cycle at 350°C for a minimum of one hour.

(5) Allow system to cool until the mask temperature is below 100°C and pressure is about 10^{-6} mm of mercury.

(6) Set substrate heaters on auto-cycle at 350°C.

(7) When 350°C is reached flash getter filaments and load internal cold trap with liquid nitrogen.

(8) Allow ten minutes to elapse.

(9) When the chamber pressure registers in the 10^{-7} mm of mercury range, the evaporation is conducted.

Normally, the pressure should rise only to a pressure of 1 to 5 x 10^{-6} mm of mercury during the evaporation.

b. Mounting

Glass stems, each with a pump-out tubulation, were fitted with either tab-clips spot welded to the leads or spring clips soldered to the leads. The stems were then cleaned in chromic acid, rinsed with distilled water, methyl alcohol, and dried with hot air.

c. Bonding

Hanovia No. 2 setting silver cement was used for more than a year as the principal bonding cement employed here. It has given excellent service except for a single poor batch. Recently DuPont No. 5504A silver loaded epoxy cement has been employed. It gives bonds of excellent strength and is presently being evaluated with regards to any effects it may have on the aging of the resonators.

d. Final Plating

Units were plated to frequency in a separate vacuum chamber using the same pumping technique described for base plating. Operating pressures were in the 10^{-6} mm Hg range. Plating of both sides simultaneously was controlled by electrically linked and maintained filament currents. The frequencies of the corrected units may be brought to within one hundred cycles (1 ppm) of the desired frequency.*

e. Final Assembly, Baking and Sealing

Bulbs were precleaned with chromic acid, rinsed in boiling distilled water, methyl alcohol and dried with hot air. The stem was sealed

*See Quarterly Report No. 3 of Contract DA-36-039-sc-85363, 1 April 1961, for more details of filament control circuit.

to the bulb on a glass sealing lathe which rotated both parts at the same angular velocity. A flame from a hand-held torch was applied to the mated parts until the seal was completed.

As shown in Figure 5 the tubulation of each envelope was inserted into a gasket-sealed packing gland leading to the vacuum chamber. In addition to the normal pumps for evacuating the chamber, four getter filaments within it were employed to lower the pressure. These were arranged to be flashed one at a time or simultaneously. A tank-type cold trap for liquid nitrogen, situated within the chamber, reduced the pressure even further (by at least one order of magnitude). The procedure for sealing resonators is given below.

(1) Evacuate the chamber.

(2) Flush it at least twice with argon.

(3) Evacuate chamber and continue pumping for one hour.

(4) Flash getters and load cold trap with liquid nitrogen. (The pressure in the chamber drops to the low 10^{-7} mm Hg range as the result of the latter operations.)

(5) Place the oven over the envelopes and bring the oven to a temperature of 175°C; hold for a minimum of three hours.

(6) Remove the oven and tip-off the units with a hand-held torch.

2. Resonators Fabricated and Measured

Polished AT-cut quartz resonator blanks of the approximate fundamental frequencies 11.1, 14.3, and 20 megacycles designed for overtone operations at 100 Mc were obtained from commercial suppliers. Over 220 of these have been plated and examined during the period of the project. The blanks were coated principally with silver, copper, aluminum, or combinations

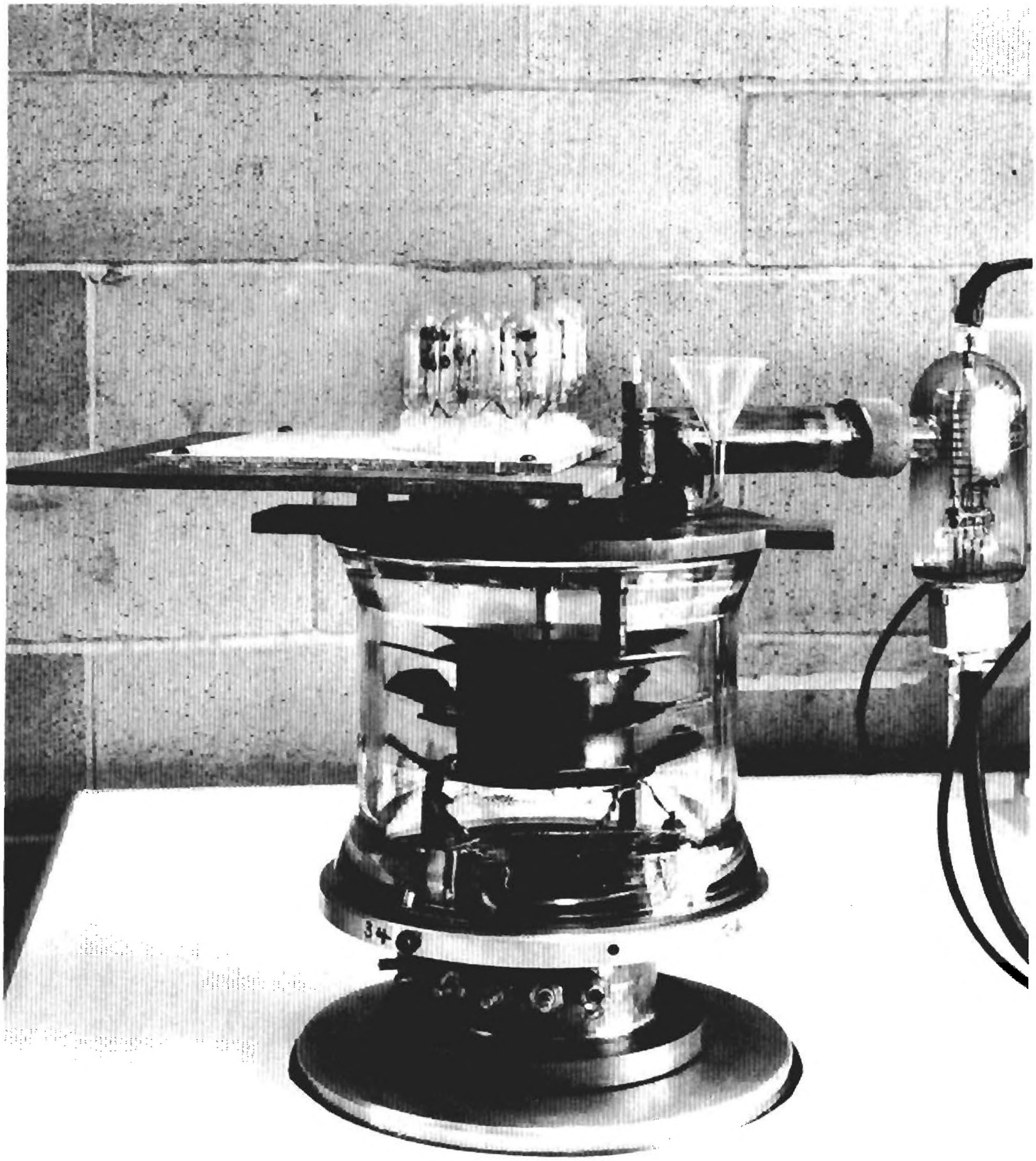


Figure 5. Vacuum baking apparatus showing mounted crystal units in position for evacuation and sealing.

thereof. More precise details concerning each resonator group are listed in Table I.

The resonators were stored initially in a constant temperature oven at 60°C. Frequency measurements were made daily for the first fourteen days and weekly thereafter. The resonators were operated only during the measurement period which normally took about 3 minutes. Drive levels were maintained as low as possible, of the order of 5 micro-watts.

Although a number of fabrication and measurement difficulties were encountered, some resonators from the beginning exhibited high stabilities. The aging data for a typical resonator (B-9-4-Ag) are shown in Figure 6. This unit was base plated with silver, with no overcoat, and operated in the ninth overtone mode as signified by the number "9" following the group designation, "B".

The graph also exhibits shifts in frequency occurring on six occasions during the period in which efforts were primarily directed toward solving the measurement problems.

Frequency data for other typical units are shown in Figure Nos. 17-34 of the Appendix.

a. Resonators Stored at 0°C

Aging data on some 15 resonators stored at 0°C have been obtained. These data are exhibited in Table II. All units included in these measurements were fifth overtone units. Aging of units of this mode has generally been relatively rapid at 60°C. However, at 0°C the frequency drift for seven units over a period of 32 days has been relatively low. The behavior of resonator K-5-8-Ag is displayed in Figure 7. In about 30 days this unit has changed in frequency only a few parts in 10^8.

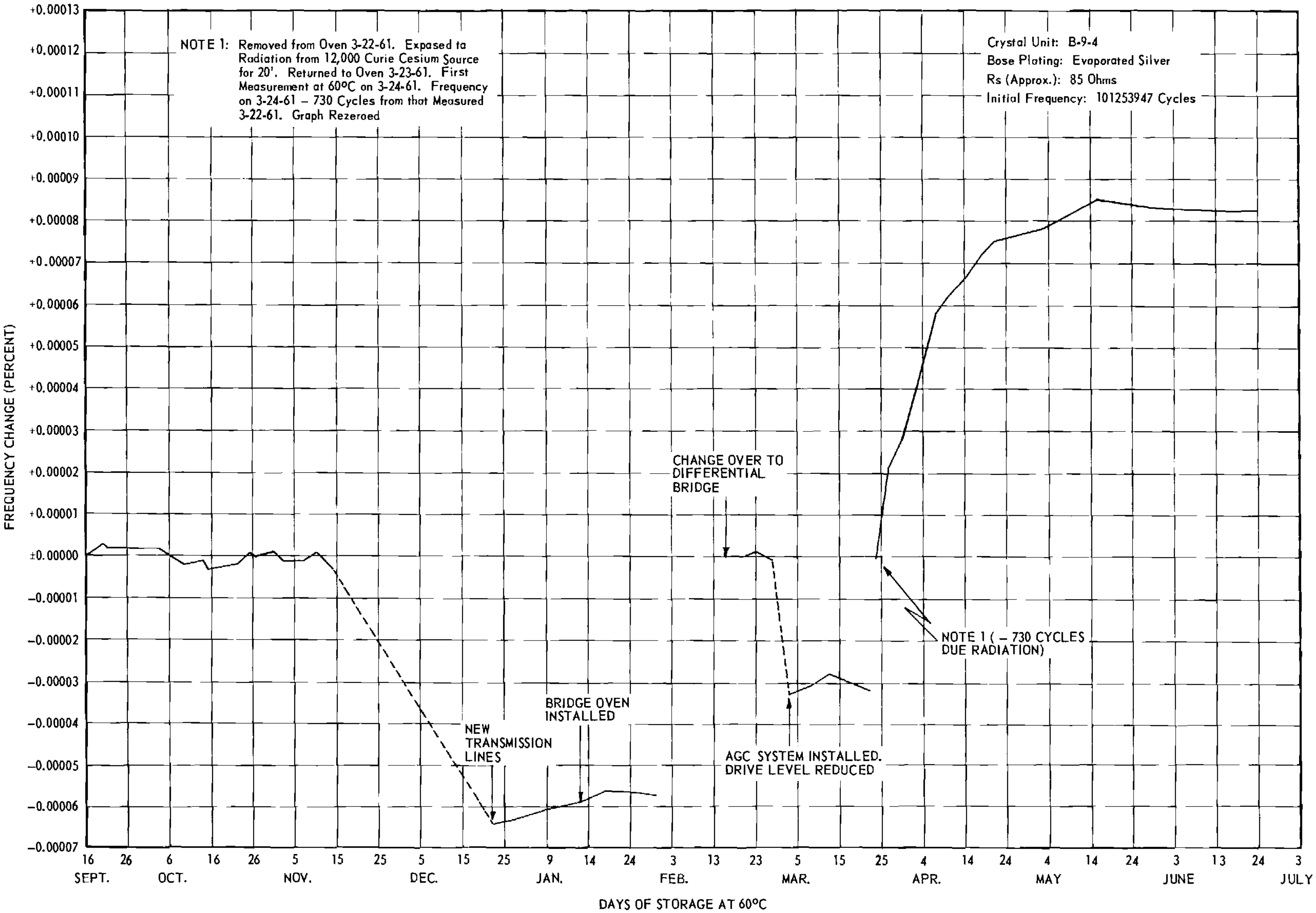


Figure 6. Frequency versus time data for resonator B-9-4-Ag, a ninth overtone unit plated with evaporated silver and stored at 60°C.

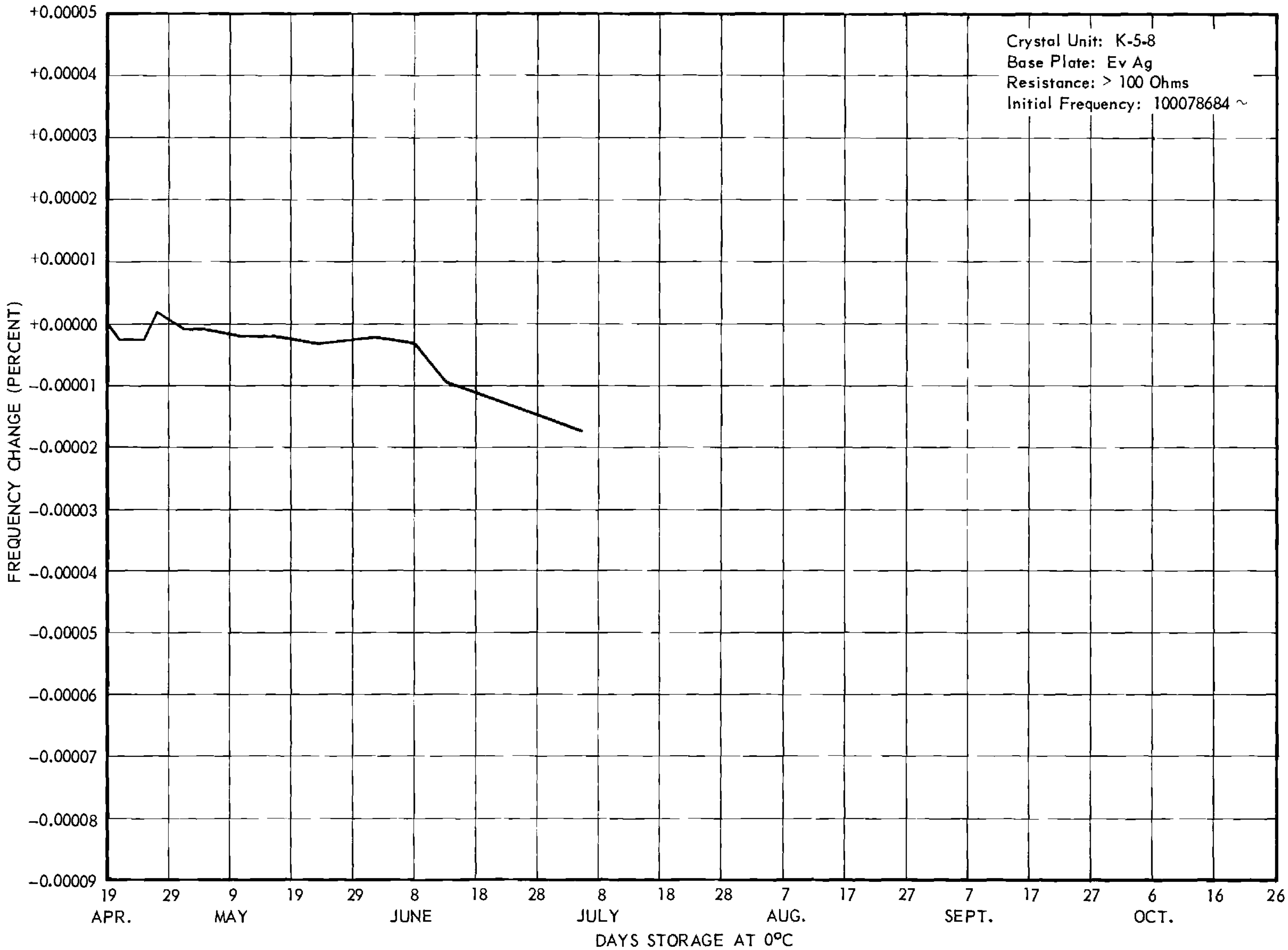


Figure 7. Frequency versus time data for resonator K-5-8-Ag, a fifth overtone unit plated with evaporated silver and stored at 0°C.

TABLE I

Parameters of 100 Mc Quartz Resonators Fabricated in the Period 1 July 1960 - 15 May 1961

Group	Over-tone	Plat-ing*	Fabri-cation Date	Initial Storage Temp.(°C)	No. Units Fabri-cated	No. Units Oper-able	Remarks
A-5	5	Al	11-11-59	60	10	6	4 of 10 rejected due low activity.
B-5	5	Al	9-19-60	60	10	6	4 of 10 rejected due low activity.
C-5	5	Al	9-26-60	60	10	7	3 of 10 rejected due low activity.
D-5	5	Al+Ag	10-11-60	60	10	3	7 of 10 rejected for various reasons.
E-5	5	Ag	12-5-60	60	10	5	
F-5(1)	5	Cu	12-12-60	-	10	0	8 of 10 bulbs cracked. 2 rejected.
F-5(2)	5	Cu	12-14-60	-	10	0	10 of 10 bulbs cracked.
F-5(3)	5	Cu	12-17-60	60	10	3	4 of 10 bulbs cracked. 3 rejected.
G-5	5	Ag	2-20-61	(5 at 60 (5 at 0	10	10	
H-5	5	Ag+Ag	3-27-61	0	10	2	3 of 10 bulbs cracked. 5 rejected.
J-5	5	Ag+Ag	3-31-61	(7 at 60 (1 at 0	10	8	1 of 10 bulbs cracked. 1 stem broken.
K-5(1)	5	Ag	4-7-61		10	0	Poor base plate.
K-5(2)	5	Ag	4-12-61	8 at 0	10	8	2 of 10 bulbs cracked during tip-off.
A-7	7	Al	8-9-61	60	10	3	7 of 10 rejected due low activity.
B-7	7	Ag	4-18-61	(5 at 60 (4 at 0	10	9	1 of 10 rejected due low activity.
C-7	7	Ag+Ag	4-21-61	(4 at 60 (3 at 0	10	7	3 of 10 rejected due low activity.
D-7	7	Ag	5-3-61	-	10	9	
A-9	9	Al	8-16-60	60	10	9	1 of 10 rejected due low activity.
B-9	9	Ag	9-12-60	60	10	5	5 of 10 rejected due low activity.
C-9(1)	9	Ag	10-27-60	-	10	0	Poor base plate.
C-9(2)	9	Ag	11-7-60	60	10	1	Poor base plate.
D-9	9	Ag	11-14-60	60	10	6	4 of 10 rejected due low activity.

* All metal plating deposited by evaporation.

TABLE I (Continued)

	No. Units Fabri- cated	No. Units Oper- able
Total Resonators by Plating Metal		
Silver	90	54
Aluminum	50	31
Copper	30	3
Silver + Silver	30	17
Aluminum + Silver	10	3

Total Resonators by Overtone of Operation

Overtone	Fabricated	Yield
5th	130	58
7th	40	28
9th	50	21

TABLE II

Aging of Resonators Stored and Measured at 0°C

Unit Identi-fication	Test Period (Days)	Total Aging (PP 10^{-7})	Series Resis-tance (ohms)	Remarks
A-5-1*	32	+ 0.0	~ 10	5th mode, Al plating
A-5-3*	32	- 0.4	33	" " " "
A-5-9*	32	- 0.3	~ 10	" " " "
A-5-10*	32	+ 0.0	19	" " " "
C-5-2*	32	+ 1.1	39	" " " "
C-5-3*	32	- 0.8	30	" " " "
C-5-5*	32	- 1.8	~ 10	" " " "
G-5-6	13	- 2.1	43	Bonding cement trouble
G-5-7	13	- 5.2	46	" " "
G-5-8	13	- 2.8	15	" " "
G-5-9	13	- 4.0	39	" " "
G-5-10	13	- 2.4	25	" " "
H-5-1	7	- 0.6	41	
H-5-2	7	- 0.3	53	
J-5-10	7	- 0.2	40	

* These units have been pre-aged at 60°C for about 120 days.

In general, the drift of all units stored at 0°C has been small. R_s values as low as 10 ohms for 5th overtone units were noted for three of seven units.

When the 0°C oven was opened and the resonators cooled to the temperature of the surrounding freezer locker (about -17°C) and, even though the temperature was reestablished at 0°C before another measurement was made, a shift in frequency for particular units was observed. This behavior, exhibited in Figure 8 (C-5-5-A1), appears to be associated with stresses exerted on the quartz crystals by the channeled tab supports used in this case, in lieu of the conventional spring clip. Such behavior was characteristic only of resonators mounted with tab clips. (See discussion of temperature cycling below.)

b. Temperature Cycling Studies

The aging rates of units were first established at either 0°C or 60°C. The units were then transferred to the temperature cycling oven* and cycled between 0 and 60°C at a rate of 5°C/hour for nine days. The units were then returned to the fixed-temperature ovens. The initial changes due to cycling and the change in the aging rate could then be established.

Table III exhibits the changes observed for 13 resonators. Figure 9 shows the aging rate of unit C-5-2 before and after cycling. Whereas the units mounted in 0.006" spring clips exhibited very small frequency changes in most instances, the frequency changes of one particular group in tab clips was very large. A correlation between the thickness of the quartz plate and the frequency changes produced by cycling also seems to exist; the thicker plates

*The design and construction of this oven and control circuit was covered in the Final Report of Contract DA-36-039-sc-78905.

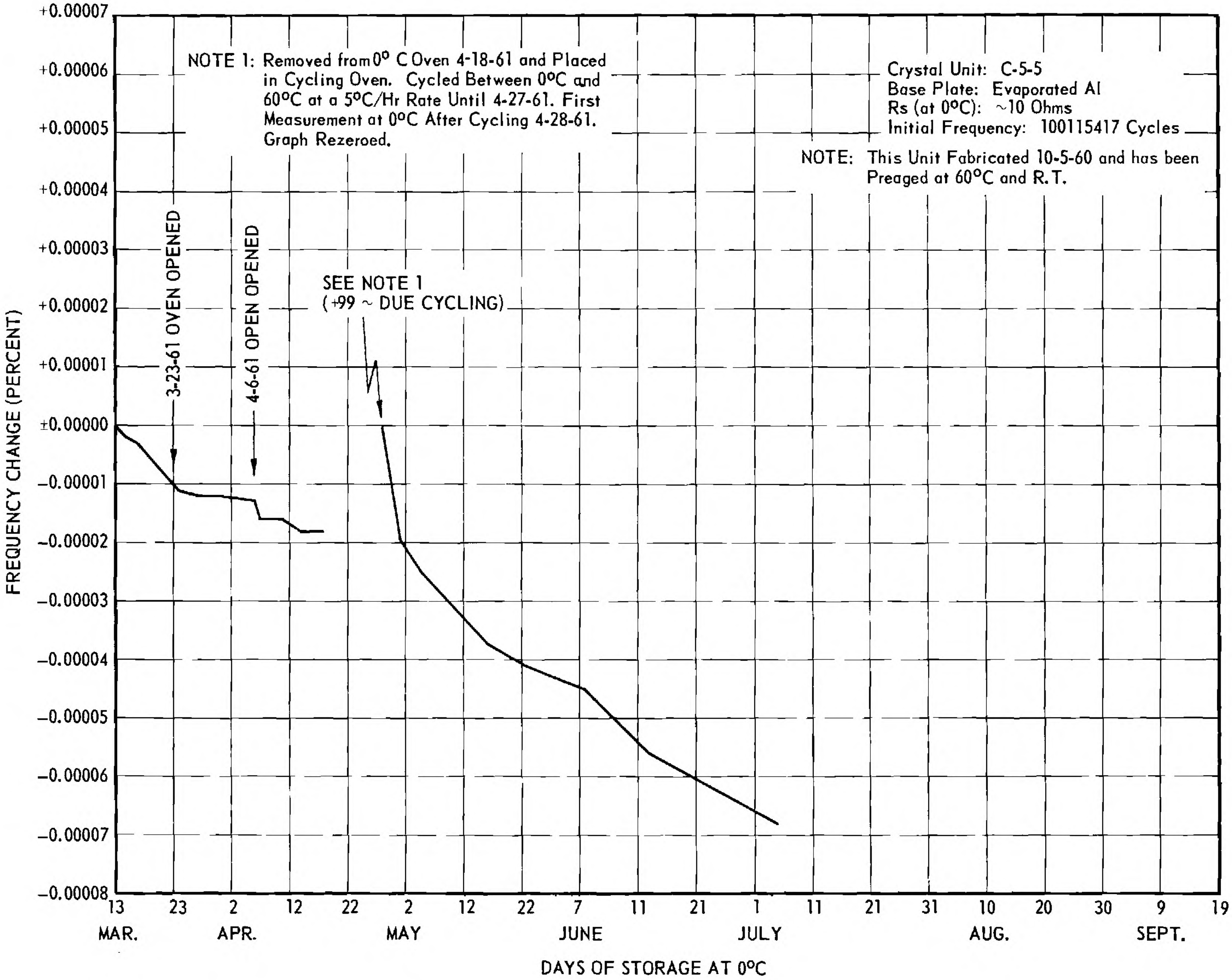


Figure 8. Aging data for resonator C-5-5-Al, an aluminum-plated, fifth overtone unit stored at 0°C. Note peculiar drops in frequency observed when oven temperature drops below 0°C during period oven is open.

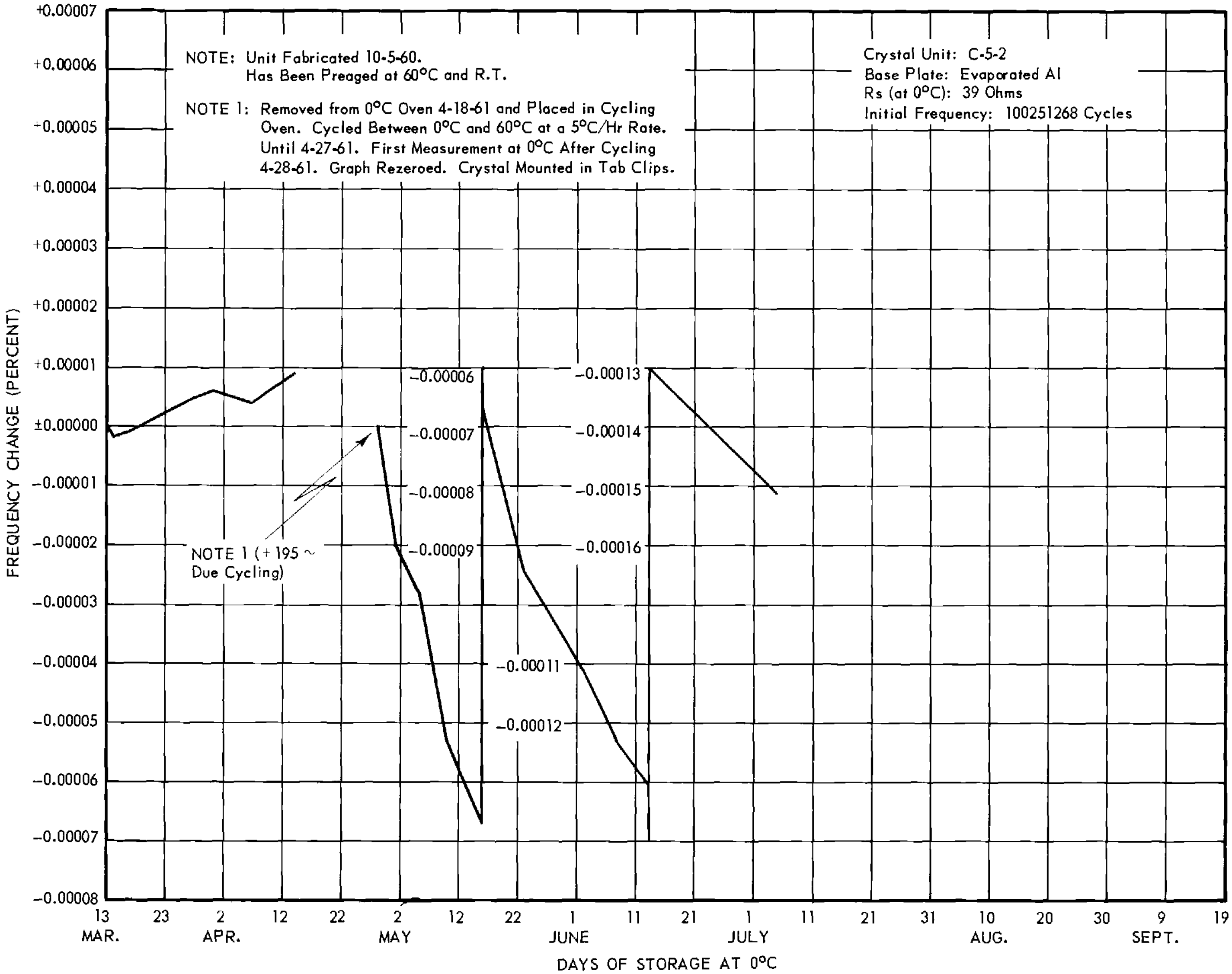


Figure 9. Aging data for resonator C-5-2-Al, an aluminum-plated, fifth overtone unit before and after temperature cycling between 0 and 60°C.

TABLE III

Effects of Daily Temperature Cycling Over the Range
0° to 60°C on 100 Mc Resonators

Unit	Over-tone	Plating	Crystal Diameter	Bonding Cement	Crystal Mount	No. Days Cycled	ΔF (cycles)*	ΔR_s (ohms)*
A-5-1	5	Al	0.375"	Hanovia No. 2	0.006" Springs	9	+ 1	N.C.
A-5-3	"	Al	"	"	"	"	+ 2	N.C.
A-5-9	"	Al	"	"	"	"	+ 1	N.C.
A-5-10	"	Al	"	"	"	"	- 3	N.C.
C-5-2	"	Al	"	"	Tab** Clips	"	+ 195	+ 2.0
C-5-3	"	Al	"	"	"	"	+ 28	N.C.
C-5-5	"	Al	"	"	"	"	+ 99	N.C.
A-5-6	"	Al	"	"	0.006" Springs	7	+ 15	N.C.
B-5-4	"	Al	"	"	Tab Clips	"	+ 14	N.C.
A-7-7	7	Al	"	"	"	"	- 11	N.C.
B-7-8	"	Ag	0.450"	DuPont 5504 A	0.006" Springs	"	+ 23	N.C.
B-9-2	9	Ag	0.460"	Hanovia No. 2	Tab Clips	"	- 5	N.C.
D-9-3	"	Ag	"	"	0.006" Springs	"	N.C.	N.C.

* Measured at storage temperature (0 or 60°C) after cycling.

** Tab clips obtained from Diebel Tool and Die Company, Chicago, Illinois.

underwent smaller frequency shifts.

D. Effects of Radiation on Quartz Resonators*

1. Introduction

One of the purposes of this research is an investigation of the effects of radiation similar to that in the Van Allen Belt on the stability of the AT-cut quartz resonators of 100-Mc frequency. In 1959 at the first nationally sponsored Symposium on the Exploration of Space, Van Allen** presented data representing intensity values in the Van Allen Belt:

1. Inner zone - 3600 km on the geomagnetic equator
    a. Electrons of energy greater than 20 kev: maximum unidirectional intensity of $2 \times 10^9/cm^2$-sec-ster
    b. Electrons of energy greater than 600 kev: maximum unidirectional intensity of $1 \times 10^7/cm^2$-sec-ster
    c. Protons of energy greater than 40 Mev: omindirectional intensity of $2 \times 10^4/cm^2$-sec
2. Outer zone - 16,000 km on the geomagnetic equator
    a. Electrons of energy greater than 20 kev: omnidirectional intensity of $1 \times 10^{11}/cm^2$-sec
    b. Electrons of energy greater than 200 kev: omnidirectional intensity of $1 \times 10^8/cm^2$-sec
    c. Protons of energy greater than 60 Mev: Omnidirectional intensity of $1 \times 10^2/cm^2$-sec
    d. Protons of energy less than 30 Mev: No significant data.

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*This section contributed in part by Dr. R. C. Palmer, Radioisotopes Laboratory of the Engineering Experiment Station, Georgia Institute of Technology.

**J. A. Van Allen, J. Geophy Res. 64, 1683 (1959).

From Range - Energy curves for charged particles*, it can be shown that the glass envelope of the resonator will stop all incident protons of energy less than ~12 Mev and all incident electrons of energy less than ~800 kev. All incident particles of higher energy will have their energy degraded by the envelope with the energy after penetration dependent on the incident energy and the thickness of the envelope. The effects of incident particles, however, are only part of the total effects which will be observed. Further radiation of the quartz crystal will result from secondary electrons, bremsstrahlung, gamma rays, mesons, and neutrons produced from the multitude of nuclear reactions that high energy protons can initiate.

An investigation of the effects of this radiation will be qualitative in nature as the only source of such varied particles is the Van Allen Belt itself. The effect to be produced by a particular type of radiation can, however, be studied quantitatively as well as qualitatively; the information gained can then serve as the basis for prediction and for investigation of the effects of the Van Allen Belt on the resonator.

Frondel** has previously made excellent studies of the effects of x-radiation on the frequencies of quartz resonators and it will be shown that the procedures used here, involving gamma rays from the Cesium-137 source, gave results which are in good agreement with the work of Frondel.

2. Resonators Exposed to Radiation from the 12,000-Curie Cesium-137 Source

Eighteen resonators whose aging behavior had been previously established by frequency measurement while stored at constant temperature were removed from the constant temperature oven and exposed to the radiation

*W. A. Aron, B. G. Hoffman, and F. C. Williams, AECU-663 (1951).

**Clifford Frondel, "Effects of Radiation on the Elasticity of Quartz," American Mineralogist 30, Nos. 1 and 2, 432 (1945).

from the Cesium-137 Research Irradiator. Levels of exposure ranged from 1×10^4 rad/hr to 1.6×10^6 rad/hr* and times of exposure ranged from ten minutes to 24 hours. The details concerning units exposed are listed in Table IV.

In general, units exposed to the greater radiation intensities at 1.4×10^6 rad/hr underwent large frequency shifts downward, in the range 3 to 10 ppm during the period of exposure. Those receiving lower intensities, 1.4×10^4 rad/hr, even for a 24-hour period shifted less than 0.5 ppm. Three units exposed a second time at high intensities underwent a somewhat lesser shift on the second exposure. All units showed rapid upward drifts when returned to the ovens. The resonators of Figures 10 and 11 exhibit typical patterns of behavior for units irradiated at high and low intensities respectively.

Additional resonators, three each from the 5th, 7th, and 9th overtone groups, have been exposed to maximum intensity radiation in the well of the Cesium-137 source. Intensities were of the order of 1.4×10^6 rad/hr. Measurements were made, at the fundamental frequency only, at intervals up to approximately 22 hours. The resonator was retrieved from the well and allowed to cool to the air conditioned room temperature for each measurement (about five minutes). The temperature in the well was approximately 10°C above room temperature. Graphs of the frequency changes with exposure time are shown in Figures 12, 13, 14, and 15; other data are given in Table V.

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*One rad is a measure of absorption of radiant energy equivalent to 100 ergs/gram mass.

TABLE IV

Parameters of 100 Mc Quartz Resonators Exposed to Radiation of Various Type, Time and Dosage

Unit Designation	Plating	Overtone	Type Radiation	Exposure Time	Dosage*	ΔF (Cycles)	ΔR_s (Ohms)	Remarks
A-9-2	Al	9	Gamma	10 min.	1.4×10^6	- 545	+ 8	Glass discolored
A-9-3	Al	9	"	"	"	- 340	+18	" "
A-9-4	Al	9	"	20 min.	"	- 527	+ 7	" "
A-9-6	Al	9	"	"	"	- 323	+ 5	" "
A-9-7	Al	9	"	30 min.	"	-1048	+16	" "
A-9-9	Al	9	"	"	"	-1097	+24	" "
B-9-2	Ag	9	"	10 min.	"	- 280	+10	" "
B-9-4	Ag	9	"	20 min.	"	- 730	Increased	" "
B-9-6	Ag	9	"	30 min.	"	- 737	Increased	Glass discolored; erratic after radiation
D-9-3	Ag	9	"	60 min.	2.0×10^4	- 29	N.C.	No visible damage
D-9-4	Ag	9	"	"	"	- 13	N.C.	" " "
D-9-5	Ag	9	"	"	"	- 18	N.C.	" " "
F-5-3	Cu	5	Proton	1 sec.	1×10^4	- 32	N.C.	" " "
F-5-4	Cu	5	"	"	"	- 1	- 2.0	" " "
D-5-1	Al+Ag	5	Gamma	24 hr.	1.4×10^4	- 16	N.C.	" " "
D-5-3	"	5	"	"	"	- 38	N.C.	" " "
D-5-4	"	5	"	"	"	- 34	N.C.	" " "
F-5-1	Cu	5	"	"	"	- 21	N.C.	" " "
H-5-1	Ag+Ag	5	"	1 hr.	1.4×10^6	- 380	+19	Glass discolored
J-5-10	Ag+Ag	5	"	"	"	- 945	+14	" "

TABLE IV (Continued)

Unit Designation	Plating	Overtone	Type Radiation	Exposure Time	Dosage*	ΔF (Cycles)	ΔR_s (Ohms)	Remarks
A-9-2	Al	9	Gamma	1 hr.	1.4×10^6	- 921	N.C.	Previously radiated 10 min. at 1.4×10^6 rad/hr.
A-9-4	Al	9	"	"	"	- 339	N.C.	Previously radiated 20 min. at 1.4×10^6 rad/hr.
A-9-7	Al	9	"	"	"	- 460	N.C.	Previously radiated 30 min. at 1.4×10^6 rad/hr.
J-5-1	Ag+Ag	5	Proton	-	-	-	-	Bulb cracked at 5μ a beam current (focused)
J-5-2	Ag+Ag	5	"	30 sec.	1×10^4	+ 50	N.C.	No visible damage
J-5-4	Ag+Ag	5	"	60 sec.	"	N.C.	N.C.	" " "
J-5-5	Ag+Ag	5	"	30 sec.	"	+ 57	- 2.0	" " "
J-5-6	Ag+Ag	5	"	"	"	+ 33	+ 7.0	" " "
J-5-8	Ag+Ag	5	"	60 sec.	"	- 28	N.C.	" " "
J-5-9	Ag+Ag	5	"	480 sec.	"	+126	N.C.	" " "

Average weight of mounted units: 10 grams

* Dose level for gamma radiation is expressed in rad/hour; one rad = 100 ergs/gram.
Dose level for proton radiation is expressed in rad/sec/μa. All radiation at 1μa.

Note: Proton radiation was at an accelerating voltage of 10^6 volts.

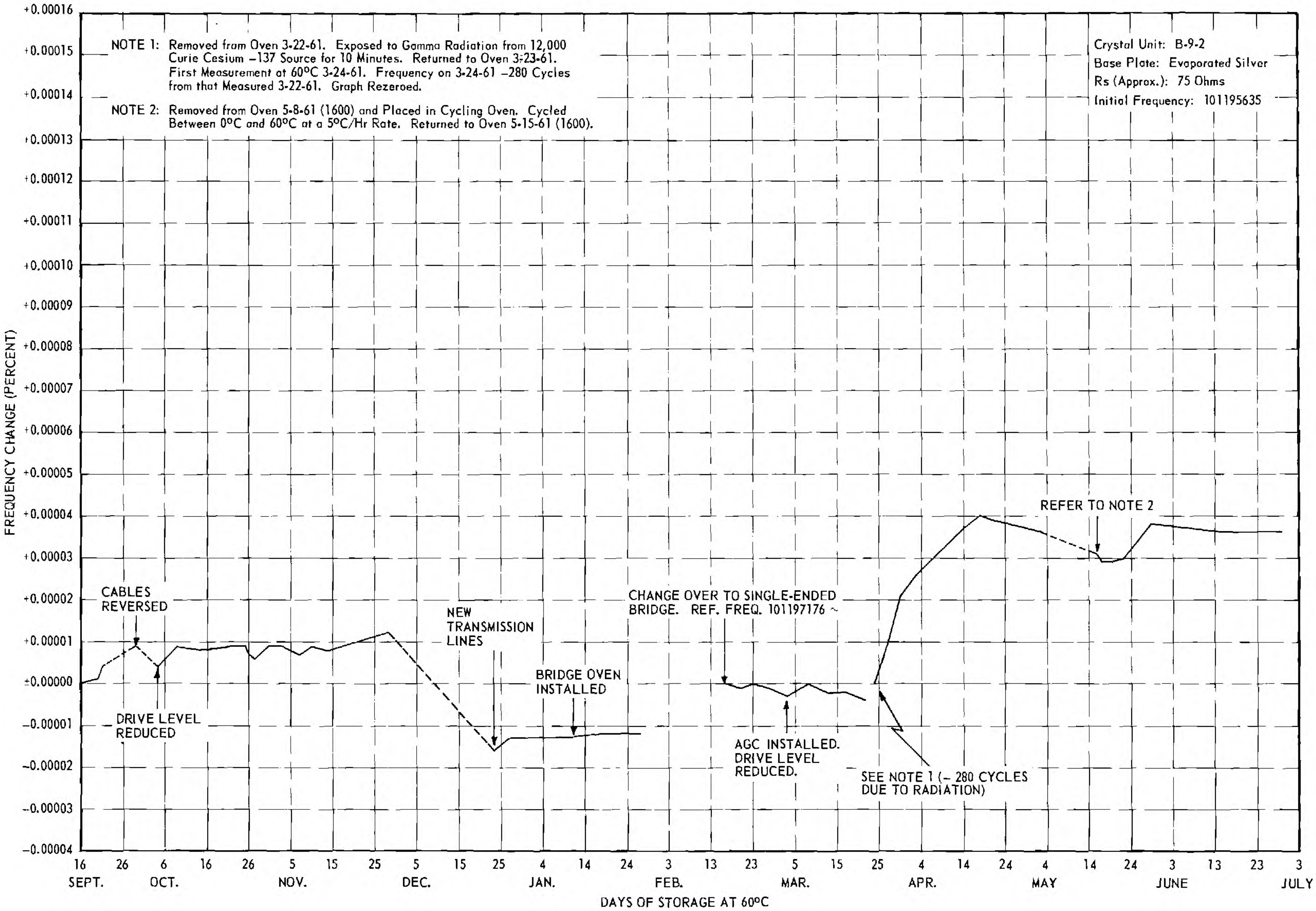

Figure 10. Aging of resonator B-9-2-Ag before and after exposure to gamma radiation from the Cesium-137 source. Dosage: 1.4×10^6 rad/hour for 10 minutes.

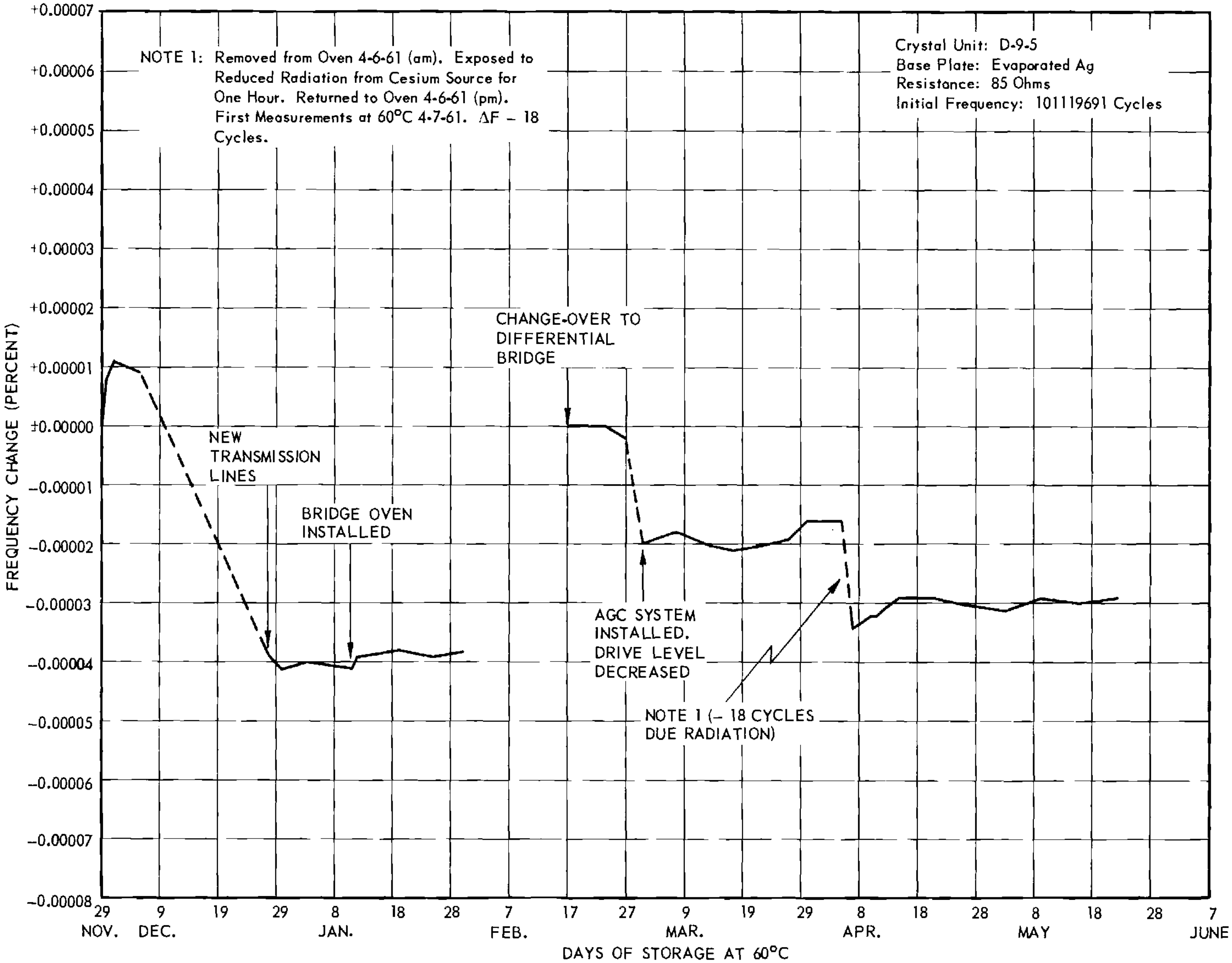

Figure 11. Aging of resonator D-9-5-Ag before and after exposure to gamma radiation from the Cesium-137 source. Dosage: 2×10^4 rad/hour for one hour.

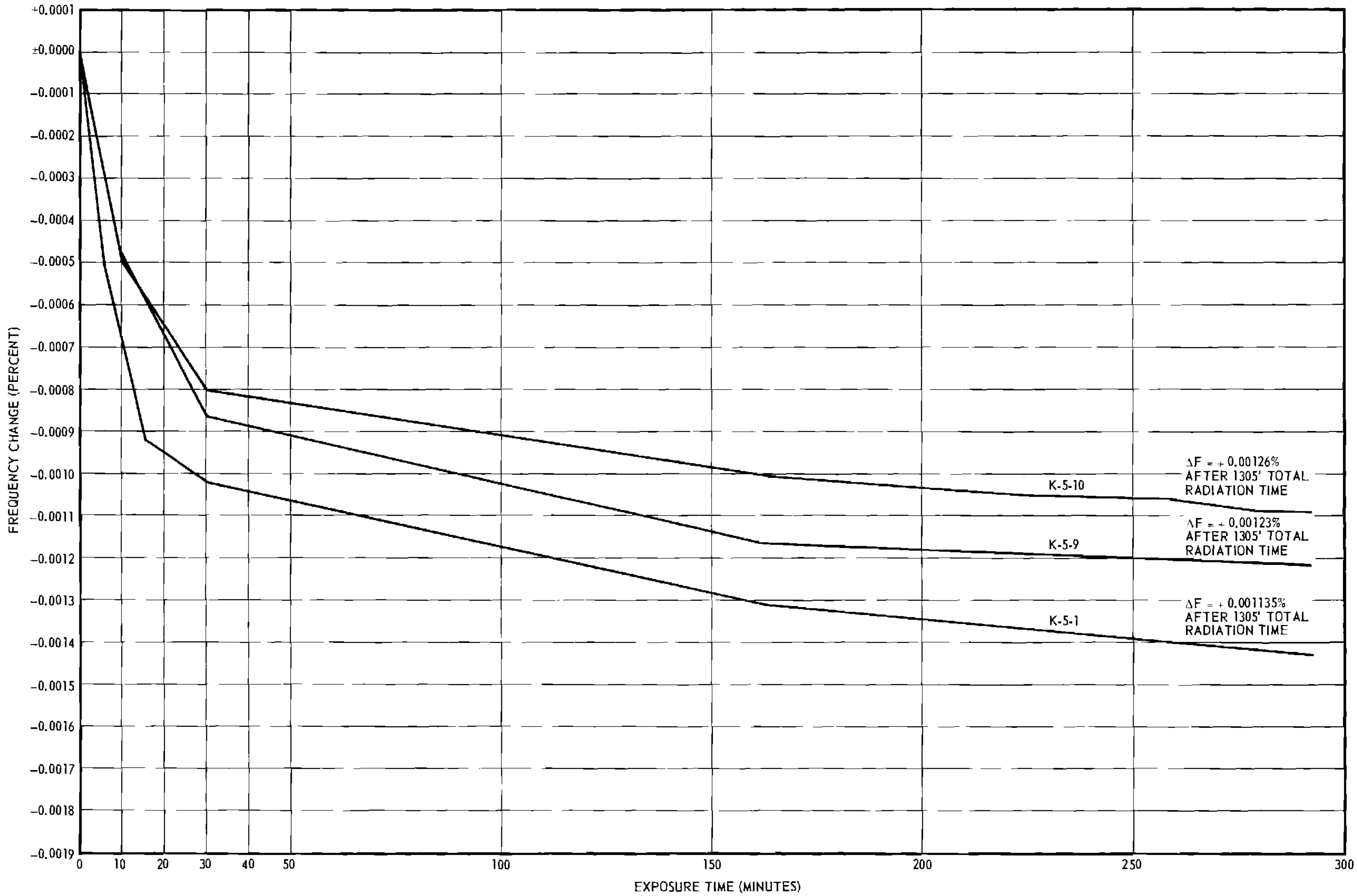

Figure 12. Frequency change with time of fifth overtone (19.9 Mc) crystal units exposed to gamma radiation of 1.4 x 10^6 rad/hour intensity.

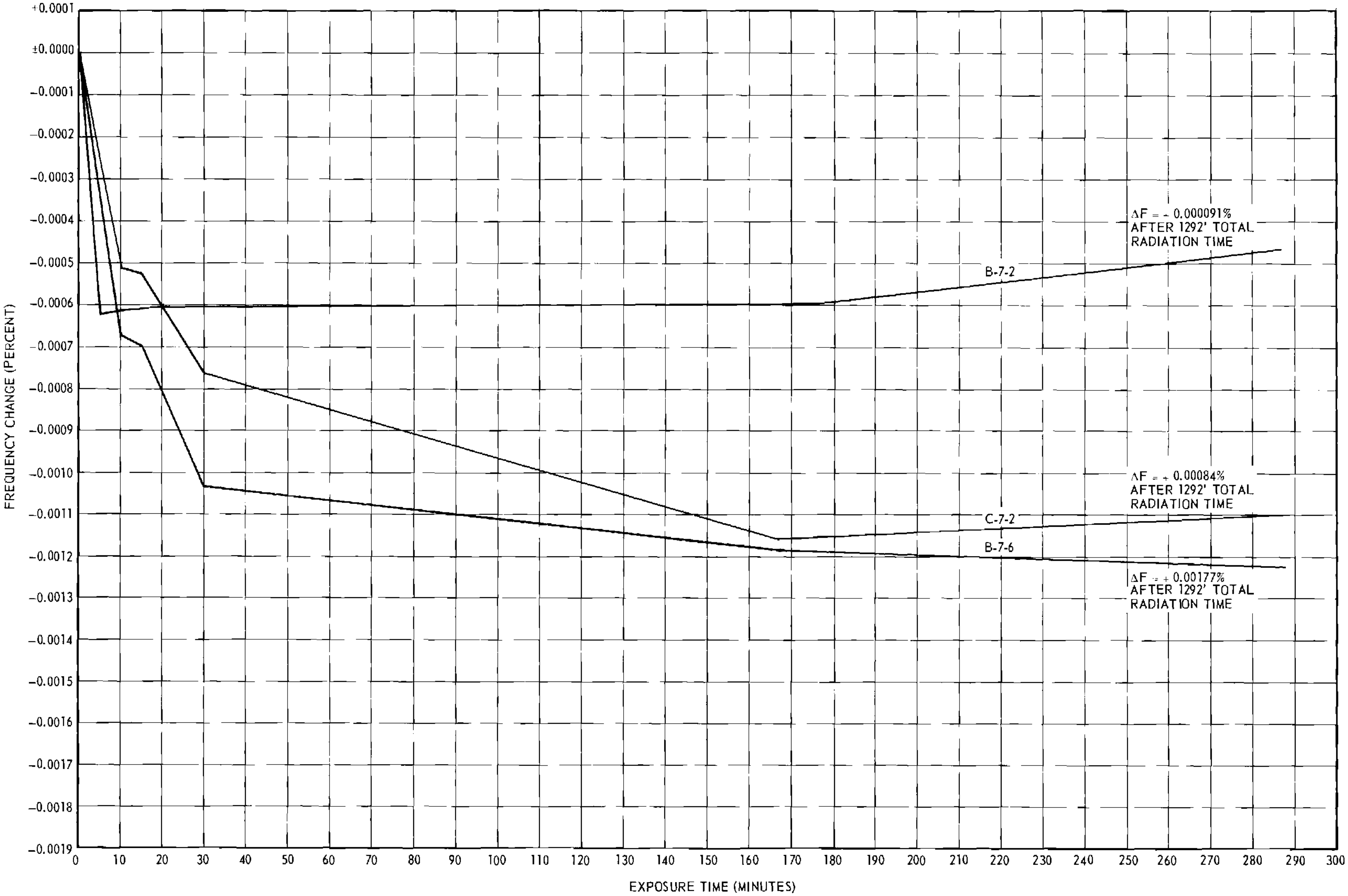

Figure 13. Frequency changes with time of seventh overtone (14.3 Mc) crystal units exposed to gamma radiation of 1.4×10^6 rad/hour intensity.

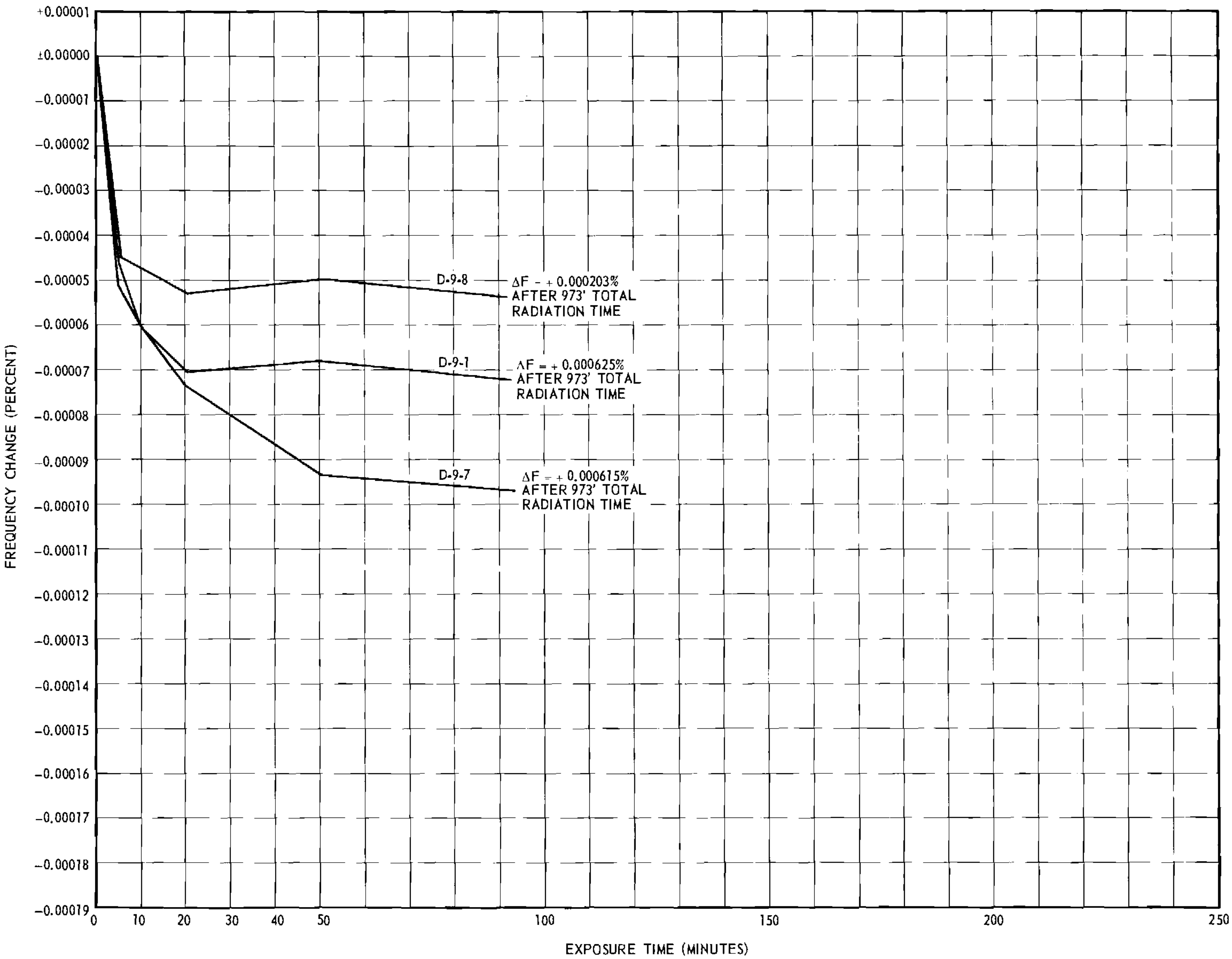

Figure 14. Frequency changes with time of ninth overtone (11.2 Mc) crystal units exposed to gamma radiation of 1.4×10^6 rad/hour intensity.

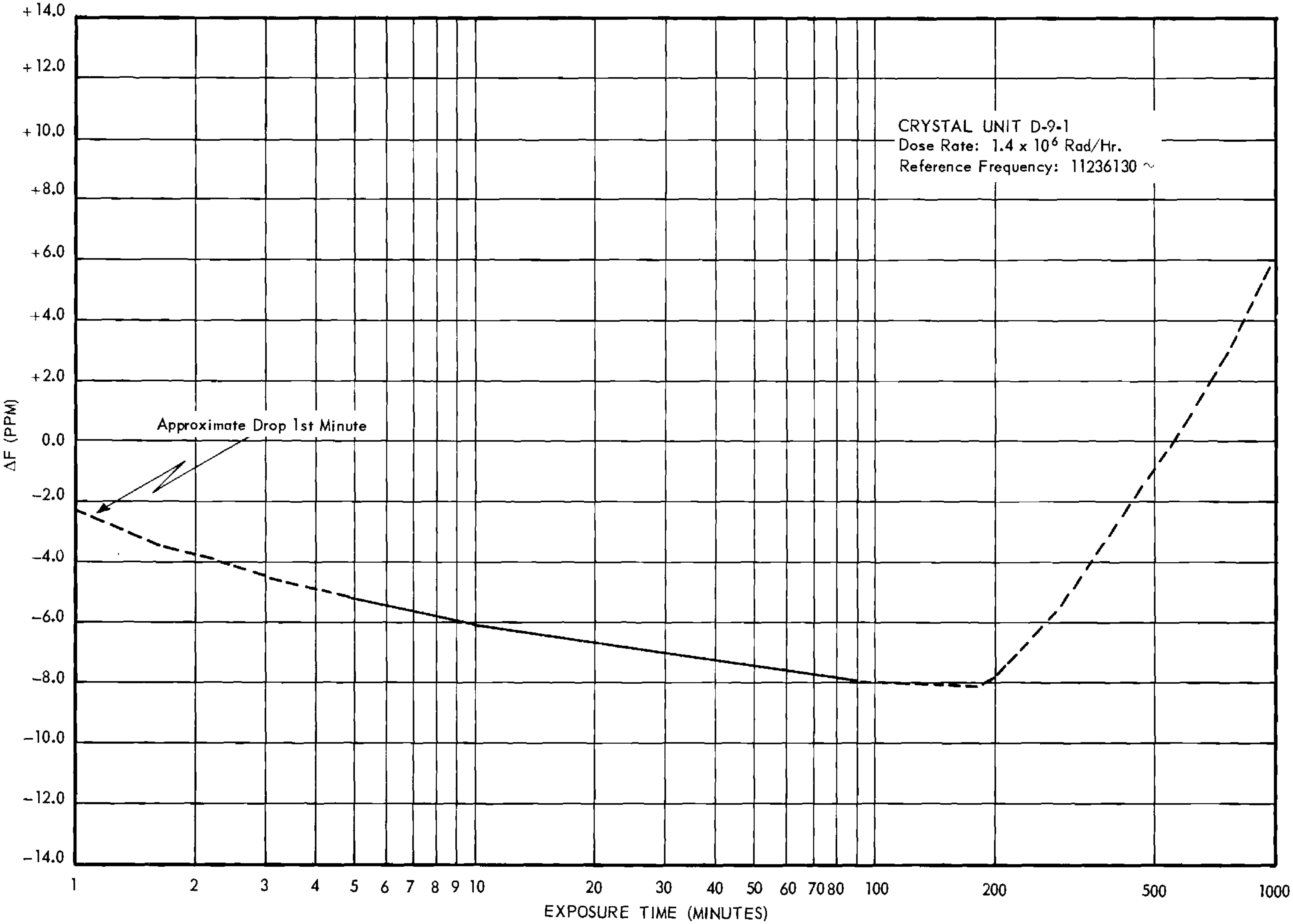

Figure 15. Typical frequency variation with Cesium-137 irradiation for resonator D-9-Ag. Note positive frequency shift after about five hours irradiation.

TABLE V

Effects of Cesium-137 Irradiation for Extended Periods on Quartz Resonators

9th Overtone Resonators

(Operated at Fundamental)

Frequency and R_s Values at Various Times

Unit D-9-1-Ag

Time of Irradiation (min)	0	5	10	20	50	90	973
Frequency Shift (cycles)	Initial Value 11, 236, 130	-58	-69	-79	-76	-81	+70
ppm	0	-5.2	-6.1	-7.1	-6.8	-7.2	+6.2
R_s Values (ohms)	4.0	4.0	4.5	-	4.0	4.0	4.5
D-9-8-Ag (Frq)	Initial Value 11, 229, 981	-50	-53	-59	-55	-60	+23
ppm	0	-4.5	-4.8	-5.3	-4.9	-5.3	+2.1
R_s (ohms)	5.5	5.5	6.0	6.0	-	-	6.0
D-9-7-Ag (Frq)	Initial Value 11, 227, 071	-52	-68	-82	-104	-108	+69
ppm	0	-4.6	-6.0	-7.3	-9.3	-9.6	+6.1
R_s	4.0	4.5	5.0	5.0	5.0	-	5.0

7th Overtone Resonators

Frequency and R_s Values at Various Times

Unit B-7-2-Ag

Time of Irradiation (min)	0	5	10	15	30	167	287	1292
Frequency Shift (cycles)	Initial Value 14, 353, 890	-89	-88	-88	-87	-87	-68	+13
ppm	0	-612	-6.2	-6.2	-6.1	-6.1	-4.8	+0.9
R_s (ohms)	5.0	5.0	6.0	6.0	5.0	5.0	5.0	5.0
B-7-6-Ag (Frq)	Initial Value 14, 341, 690	-58	-97	-99	-134	-155	-162	+120
ppm	0	-4.1	-6.8	-7.0	-9.4	-10.9	-11.3	+8.4
R_s (ohms)	5.0	5.0	5.5	5.5	5.5	5.0	-	5.0
C-7-2-Ag (Frq)	Initial Value 14, 264, 906	-38	-74	-75	-109	-153	-144	+254
ppm	0	-2.7	-5.2	-5.3	-7.6	-10.6	-10.1	+17.7
R_s	4.5	4.5	5.0	5.0	-	4.5	-	5.0

TABLE V (Continued)

5th Overtone Resonators

Frequency and R_s Values at Various Times

Unit K-5-1

Time of Irradiation (min)	0	5	10	15	30	162	292	1305
	Initial Value							
Frequency Shift (cycles)	19, 978, 766	-91	-133	-183	-208	-263	-286	+252
ppm	0	-4.6	-6.7	-9.2	-10.4	-13.1	-14.3	+12.6
R_s	5.0	5.5	6.0	6.0	-	6.0	5.5	5.5
	Initial Value							
K-5-9-Ag (Frq)	19, 980, 263	-61	-96	-115	-172	-232	-244	+247
ppm	0	-3.1	-4.8	-5.8	-8.6	-11.6	-12.2	+12.3
R_s	5.0	5.5	5.5	5.5	5.0	5.0	5.5	5.5
	Initial Value							
K-5-10-Ag (Frq)	19, 992, 261	-62	-98	-116	-160	-200	-219	+227
ppm	0	-3.1	-4.9	-5.8	-8.0	-10.0	-10.9	+11.4
R_s	5.0	5.5	5.0	-	5.0	5.0	5.0	-

Note: Resonators were exposed to Cesium-137 radiation at 1.6×10^6 rad/hour. They were retrieved from the well and allowed to cool five minutes (to air conditioned room temperature for each measurement).

It is worthy of note that the largest negative frequency shift occurs in the first few minutes and that thereafter the slope is much smaller or non-existent. After several hours the frequencies begin to rise again. For all nine resonators a plus value was indicated for the final reading of 17 to 22 hours. This was of the magnitude of +1 to +18 ppm. In general, the 7th and 9th overtone resonators exhibited smaller shifts than the 5th overtone resonators. R_s values exhibited relatively small changes.

No measurements were completed on post-saturation-dose frequency drifts. This measurement appears to be a highly desirable one since it would reveal whether radiation pretreatment of resonators would be a condition for maximum stability under subsequent radiation.

3. Resonators Exposed to the 1-Mev Proton Beam of the Van de Graaff Generator

Nine resonators were exposed to the 1-Mev proton beam of the Van de Graaff generator. These were located so that the beam impinged at the top of the glass envelope of the resonator and was directed generally along the axis of the envelope. The beam was set at approximately one microampere and times were varied between one and 480 seconds. Dosage was calculated at approximately 1.0×10^4 rad/sec-microamperes of beam current.

Four of the nine units registered small positive frequency shifts, two no change, two small negative shifts, and the envelope of one was cracked in the beam. Shifts were under 1 ppm except in one instance.

The behavior typical of these units is indicated in Figure 16.

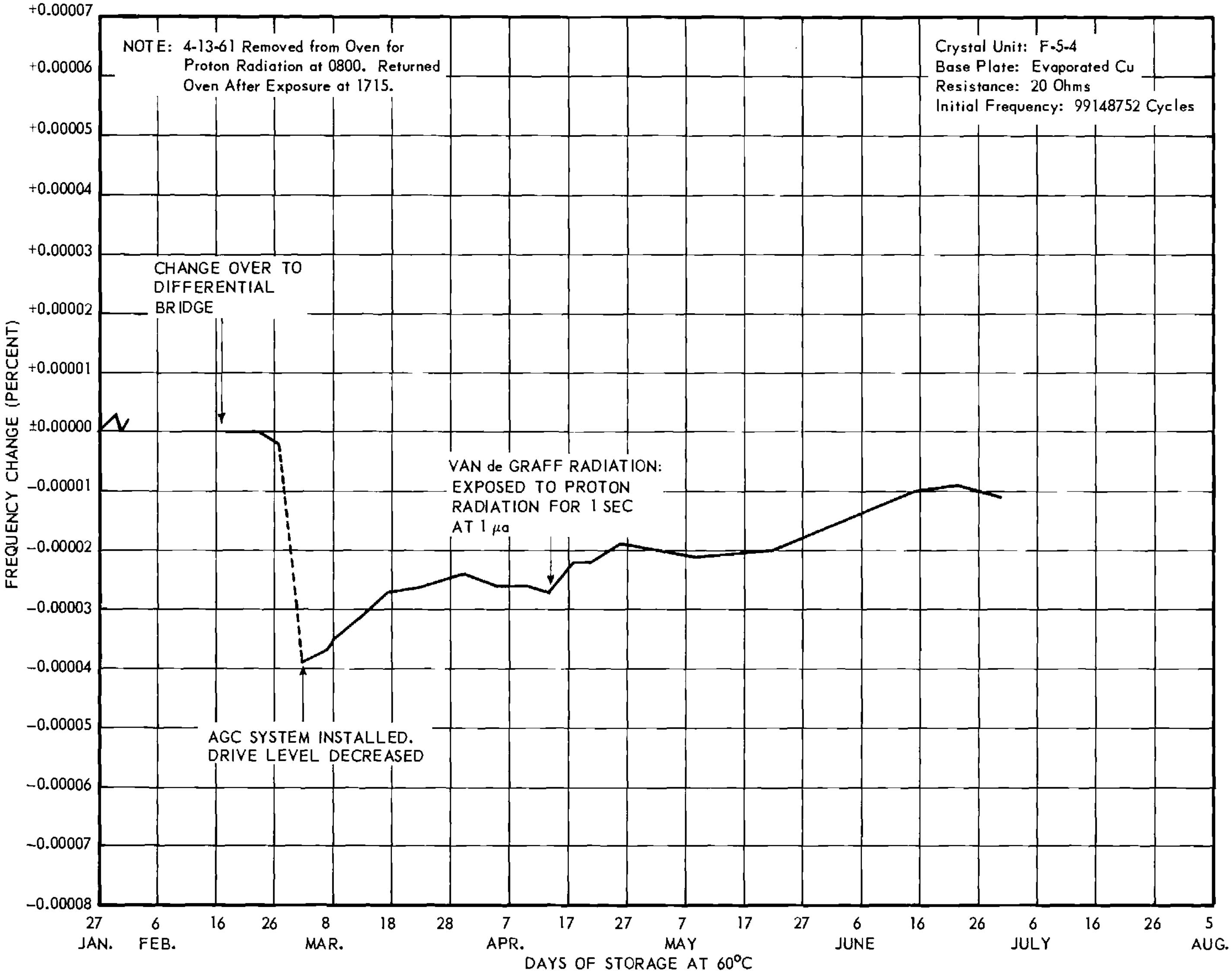

Figure 16. Aging of resonator F-5-4-Cu before and after exposure to proton radiation of 1.0 x 10^4 rad/sec/microampere of beam current intensity for one second at one microampere.

V. DISCUSSION

Measurement of the frequency to the desired accuracy was recognized at the outset as a difficult problem. The development of a method of driving the resonators and measuring their frequencies, in a few minutes, to ±2 parts in 10^8 is considered by the authors to be a singular achievement.

It was evident early in the work that units of the ninth overtone exhibited superior stability; the aging of 13 of 18 units was less than three parts in 10^7 during the period of test and five under 1.5 parts in 10^7. Seven of these were in test at 60°C for approximately nine months and five for six months. Units operated at the fifth and seventh overtones were somewhat less reliable but a number of units of each group exhibited stabilities equivalent to that of the ninth overtone units.

Although the projected evaluation of metal coatings and combinations during the period was not completed, it was apparent that silver alone was an excellent coating and that the use of both aluminum and copper as base-coating materials was feasible. Insufficient data were obtained to evaluate the effect on stabilities of overcoating to frequency with a second layer of the same or of a different metal.

Limited tests indicated that the drift at 0°C was markedly less than at 60° C.

The effects of temperature cycling over the range 0°C to 60°C once per day at about 5°C per hour had no effect on the stabilities of the cycled units in some cases and large effects in others. The fact that the most of the affected units were mounted in tab clips instead of spring clips suggested that thermal expansion and contraction of the mount may be undesirably stressing the resonator. This interpretation, however, is subject to further study.

Exposure of the resonators to gamma radiation from the Cesium-137 Irradiator resulted in downward shifts in frequency in accordance with the intensity and time of exposure. The larger shifts experienced were about 10 ppm. On a second similar exposure shifts were somewhat less. All resonators experienced upward recovery drifts at fairly rapid rates upon restoration to proper oven positions. Subsequent exposure to high intensity (1.4×10^6 rad/hr) for periods up to 24 hours resulted in overall positive shifts indicating a complete saturation. Unfortunately the termination of the project prevented completion of post-irradiation aging studies.

Exposure of the resonators to proton beam bombardment (1 Mev) for periods up to four minutes generally resulted in small positive shifts (a few parts in 10^7) in frequency. This behavior is ascribed to possible clean up of residual gases (within the resonator envelope) by a glow discharge evoked by the impinging beam. It is noteworthy that the direction of these shifts was opposite to those resulting from short term exposure to gamma radiation.

VI. CONCLUSIONS AND RECOMMENDATIONS

Quartz resonators of 100-Mc frequency can be fabricated to maintain stabilities better than three parts in 10^7 per year. Selected units will meet the projected requirement of 1 part in 10^7 per year.

Silver, aluminum and copper appear to be satisfactory plating materials, silver being superior. Greater stability is obtained with resonators of the high overtone values (i.e., lower fundamental frequencies) within the limits of tolerable series resistance values.

Temperature cycling effects in the range 0° to 60°C may have severe effects on resonator stability; mounting structures should be designed to eliminate or minimize the possibility of undesirably stressing the resonator plate as a result of thermal changes.

Resonator damage due to gamma radiation emitted as a result of high energy electron collision with a satellite or the resonator container will result in downward shifts in frequency. Magnitudes of 1 or 2 parts in 10^7 per day were experienced in a 24-hour period at a level simulating that in the Van Allen Belt. A saturation effect, however, exists which should be further explored as a possible method of minimizing frequency changes due to this effect.

Proton (1 Mev) bombardment, on the other hand, resulted in upward frequency shifts of a few parts in 10^7 in periods of 30 seconds to four minutes at an intensity of 1.0×10^4 rad/hr. These shifts were counter to, and compensated in part for, the downward shifts due to gamma radiation.

The early portion of the work was beset with measurement difficulties until completion of satisfactory equipment for rapid accurate measurement to one part in 10^8. More recent developments indicate the approach to a measurement accuracy near one part in 10^9. A series of leaking stems severely reduced the yield of good resonators. The correction of these matters and more recent measurements indicate that much better aging information can be obtained in subsequent work.

It is recommended that this research be continued for at least a period of 12 months and that major effort be directed toward:

(1) A more definite evaluation of coating-to-frequency with an overcoating layer of the same or a second metal.

(2) Delineation of the frequency behavior of resonators subjected to a saturation radiation dose similar to that experienced by unit D-9-1 in Figure 15.

(3) Measurement of change in frequency of units subjected to bombardment by high energy protons (30 to 40 Mev).

VII. PERSONNEL

The principal personnel employed on this project and the time devoted to it by each are noted below.

Individual	Position	Hours
R. B. Belser	Project Director	725
W. H. Hicklin	Assistant Research Engineer	1764
R. C. Palmer	Research Assistant Professor	120
W. B. Warren	Research Engineer	426
D. W. Robertson	Research Engineer	74
S. D. Witt	Research Engineer	88
J. O. Darnell	Research Assistant	1725
W. C. Knapp	Technician	160
C. M. Shirley	Technician	966
C. S. Wilson	Technician	618
W. D. Dawson	Student Assistant	618

Mr. Belser, M.S., Physics, has worked in the field of quartz crystal fabrication and measurement for 11 years. Mr. Hicklin, graduate of Valparaiso Technical Institute, has assisted Mr. Belser in all facets of this work for nine years. Radiation tests were conducted under the supervision of Richard C. Palmer, Research Assistant Professor in the Radioisotopes Laboratory. Mr. Warren and Mr. Witt, with M.S. degrees in electrical engineering and additional graduate work, have worked in the general fields of communications, electrical circuitry, electrical measurements, and instrument design for over 10 years and are project directors of related research. Messrs. Knapp, Shirley, Darnell and Dawson are skilled assistants familiar with electronic, mechanical, and vacuum technology.

APPENDIX

Resonators: A-5-1 to A-5-10

Blanks: 0.375" diameter, polished, etched, 20.09 Mc

Base Plate: 1500 A evaporated aluminum at 250°C substrate temperature

Final Plate: None

Mounting: G. E. stems with 0.006" spring clips

Bonding: Hanovia No. 2 cement

Vacuum Baking: 180°C for three hours

Comments: Crystal mounted parallel to base. Yield: 70%. This group of units was stored at room temperature for a period of approximately 8 months before data were taken for these particular plots. Good stability at 0°C. A number of the units held ±0.03 ppm for 90 days during examination.

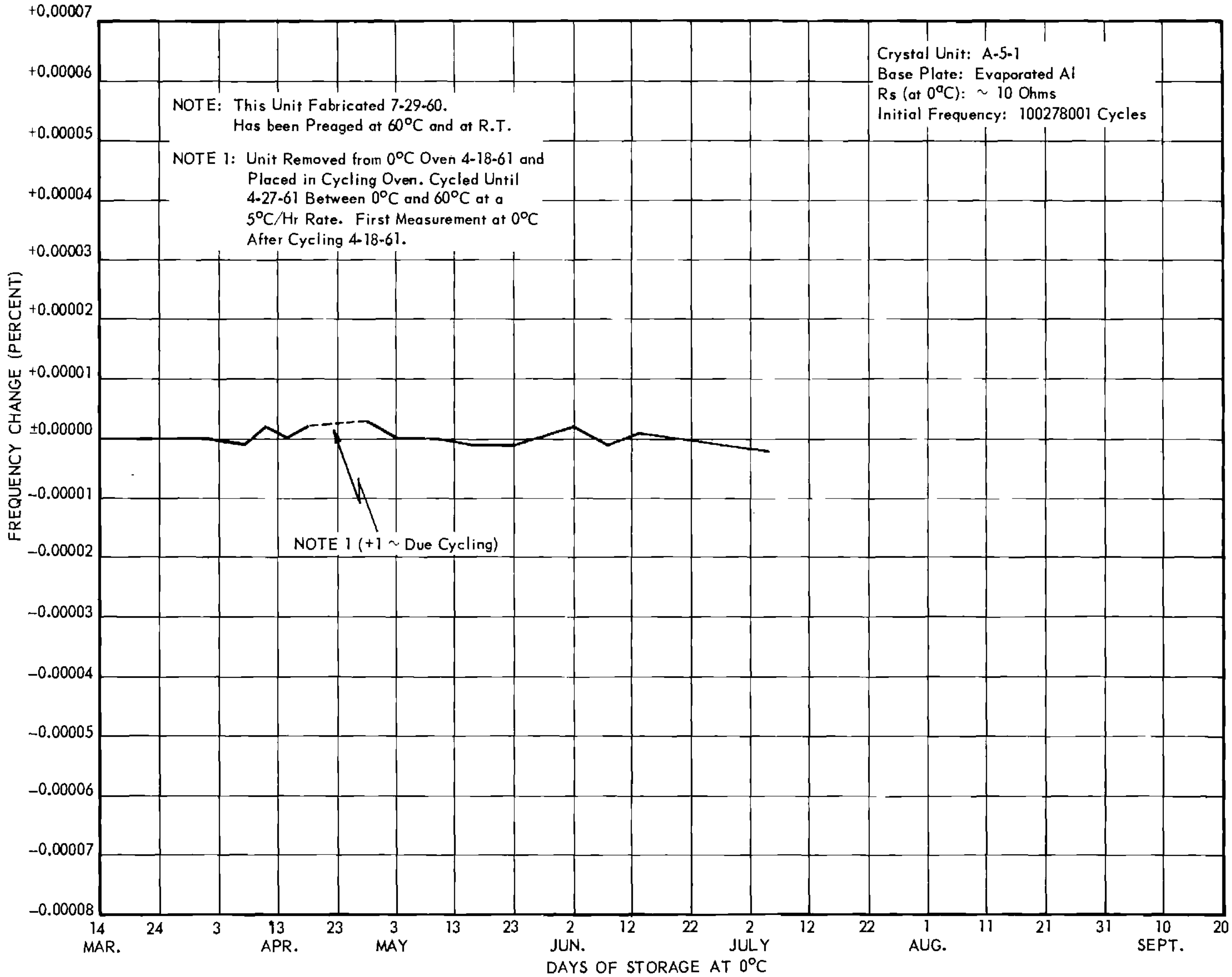

Figure 17. Frequency versus time data for resonator A-5-1-Al, a fifth overtone unit plated with evaporated aluminum and stored at 0°C.

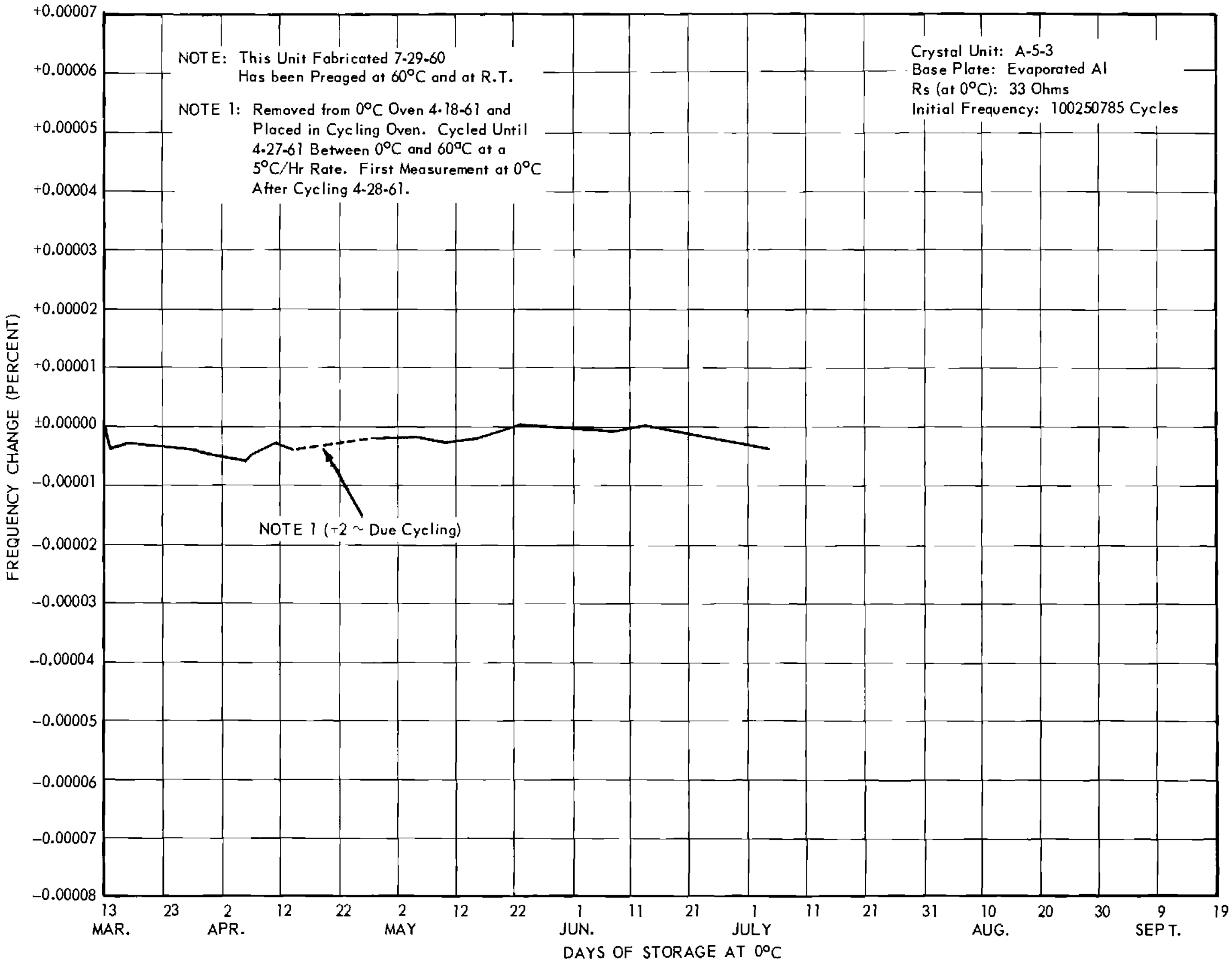

Figure 18. Frequency versus time data for resonator A-5-3-Al, a fifth overtone unit plated with evaporated aluminum and stored at 0°C.

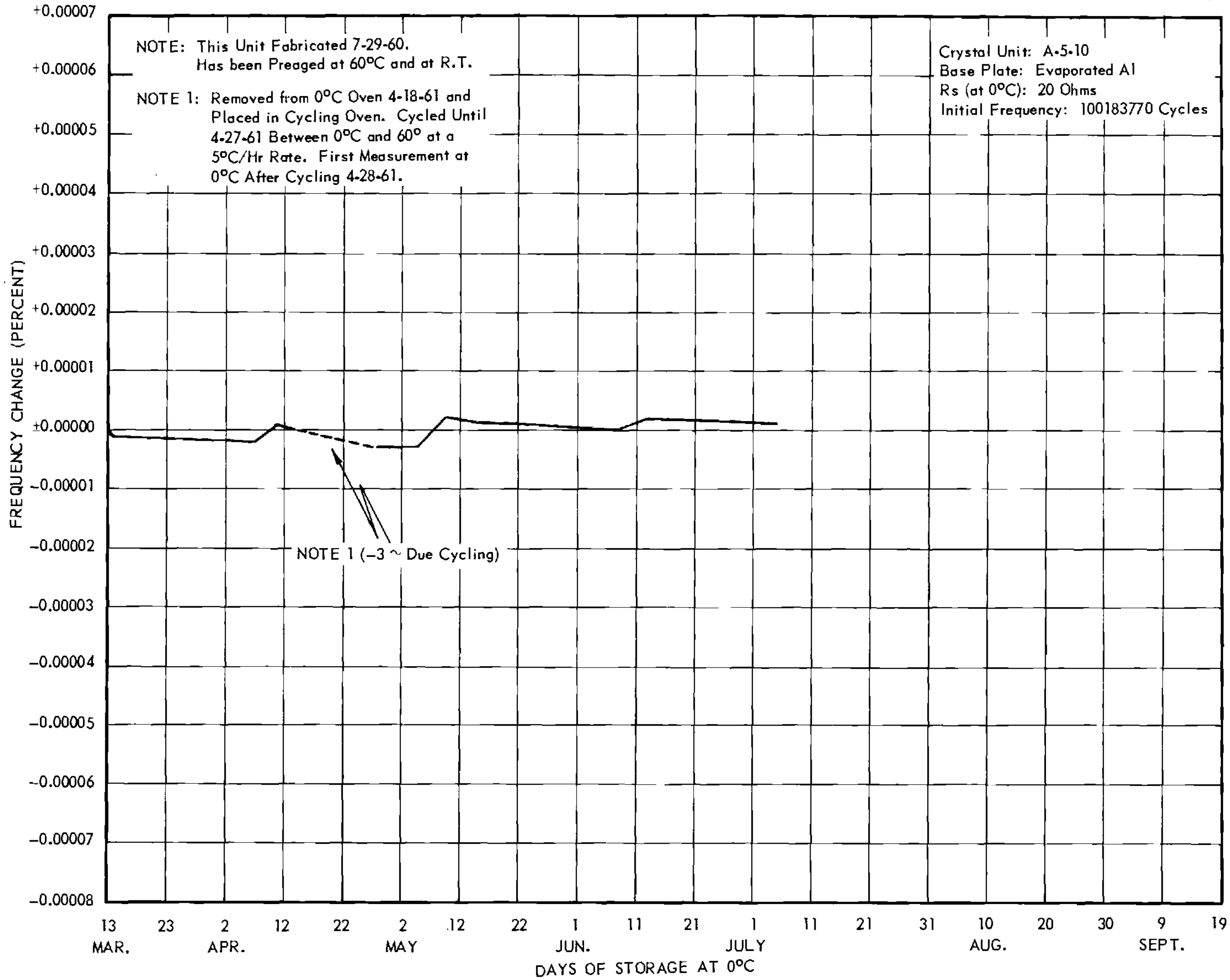

Figure 19. Frequency versus time data for resonator A-5-10-Al, a fifth overtone unit plated with evaporated aluminum and stored at 0°C.

Resonators: D-5-1 to D-5-10

Blanks: 0.375" diameter, polished, etched, 20.09 Mc

Base Plate: 1500 A evaporated aluminum at 250°C substrate temperature

Final Plate: Evaporated silver at average plate back of ~200 kc

Mounting: W.R.W. stems with tab clips

Bonding: Hanovia No. 13 cement

Vacuum Baking: Yield: 30%. Trouble sealing bulbs to new stems. These units exhibited rapid down drifts in many instances apparently due to leaks developed about lead-ins before or during sealing.

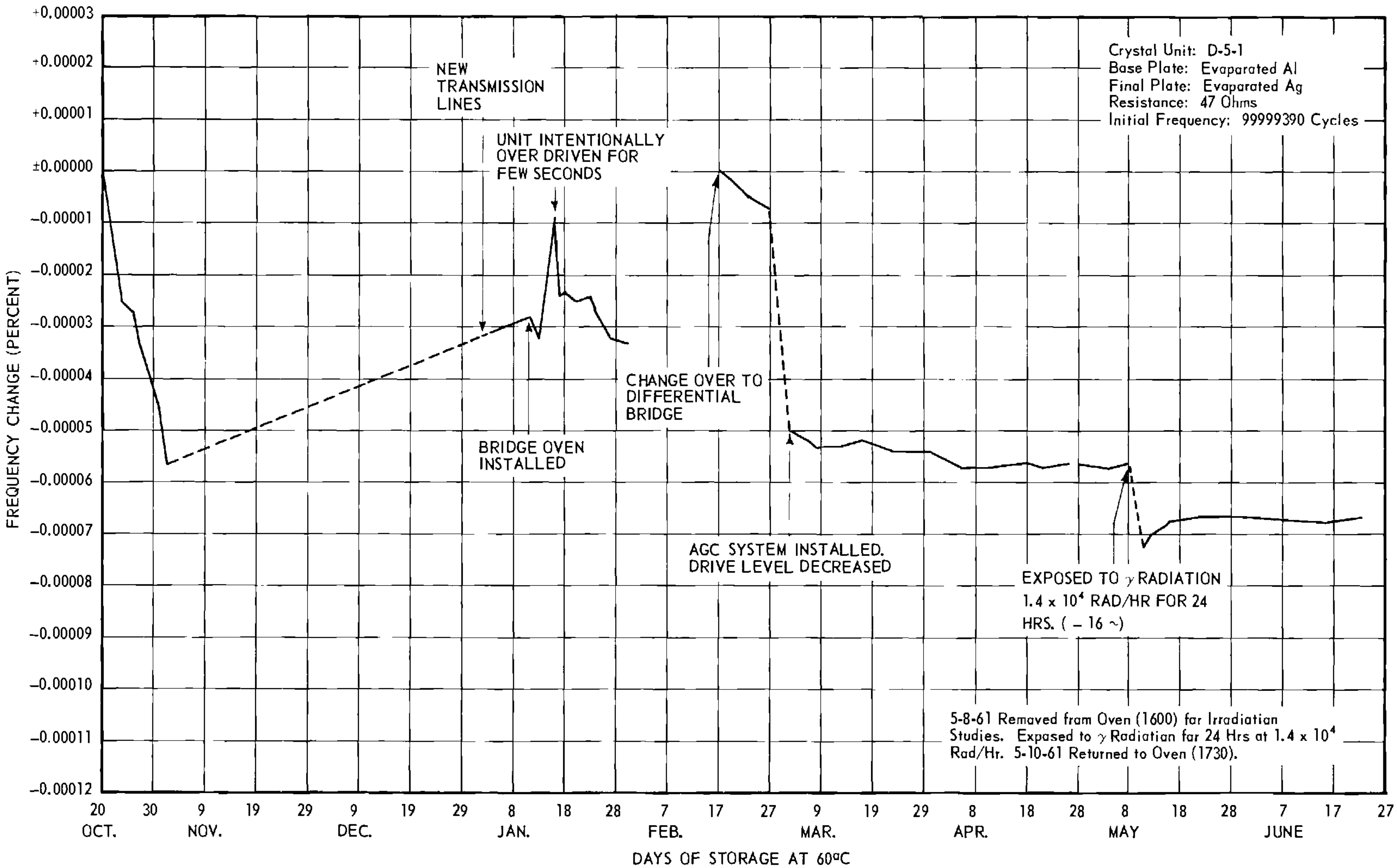

Figure 20. Frequency versus time data for resonator D-5-1-Al+Ag, a fifth overtone unit plated with evaporated aluminum plus silver and stored at 60°C.

Resonators: F-5-1 to F-5-10

Blanks: 0.375" diameter, polished, etched, 20.09 Mc

Base Plate: 1500 A evaporated copper at 250°C substrate temperature

Final Plate: None

Mounting: W.R.W. stems with 0.006" spring clips

Bonding: Hanovia No. 2 cement

Vacuum Baking: 200°C for six hours

Comments: Yield: 30%. The poor yield appears to have been related to difficulty with stem sealing. Stems also leaked about lead-ins. One sealed unit (F-5-4) exhibited exceptional stability which appears to be the true behavior obtainable with copper films (See Figure 16).

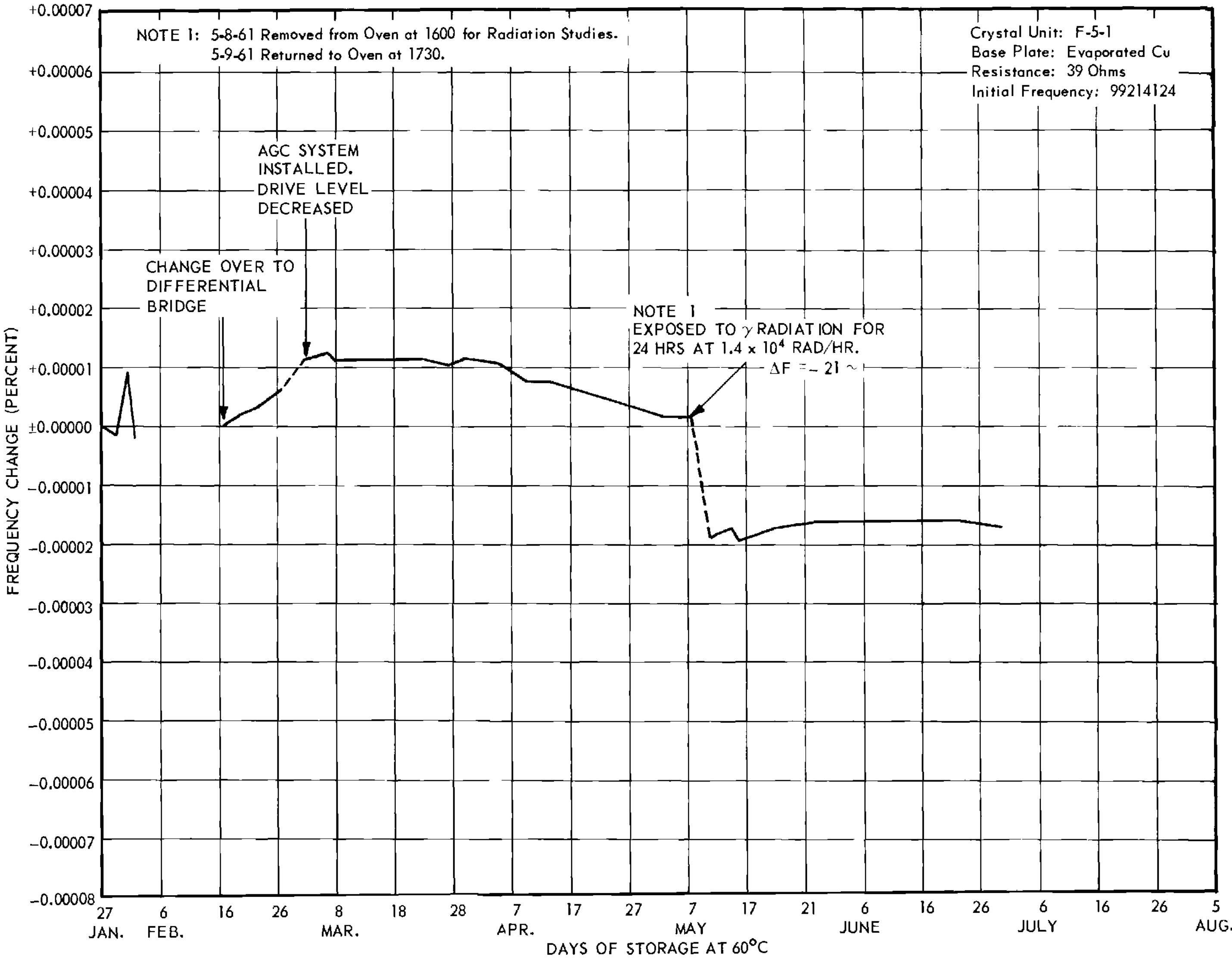

Figure 21. Frequency versus time data for resonator F-5-1-Cu, a fifth overtone unit plated with evaporated copper and stored at 60°C.

Resonators: H-5-1 to H-5-10

Blanks: 0.450" diameter, polished, etched, 20.200 Mc

Base Plate: 1500 A evaporated silver at 250°C substrate temperature

Final Plate: Evaporated silver. Average plate back: 300 kc

Mounting: W.R.W. stems with 0.006" spring clips

Bonding: Hanovia No. 2 cement

Vacuum Baking: 200°C for six hours

Comments: Yield: 20%. Extremely poor yield due to difficulty with stem sealing and leaking seals. Only one good unit.

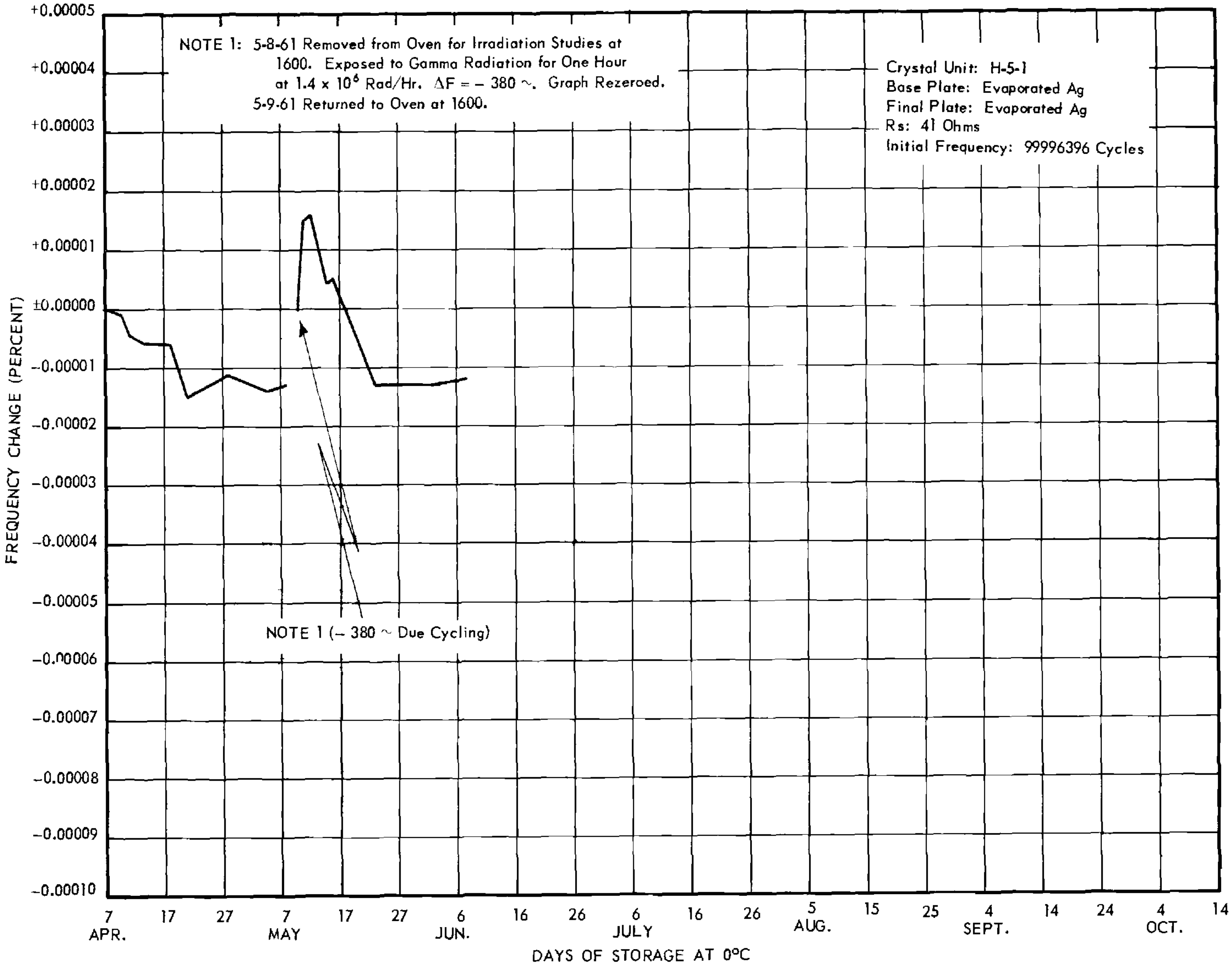

Figure 22. Frequency versus time data for resonator H-5-1-Ag+Ag, a fifth overtone unit plated with evaporated silver plus silver and stored at 0°C.

Resonators: J-5-1 to J-5-10

Blanks: 0.450" diameter, polished, etched, 20.200 Mc

Base Plate: 1500 A evaporated silver at 250°C substrate temperature

Final Plate: Evaporated silver. Average plate back: 125 kc

Mounting: W.R.W. stems with 0.006" spring clips

Bonding: Hanovia No. 2 cement

Vacuum Baking: 175°C for three hours

Comments: Yield: 70%. These units were quite erratic, with a large number of apparent leakers.

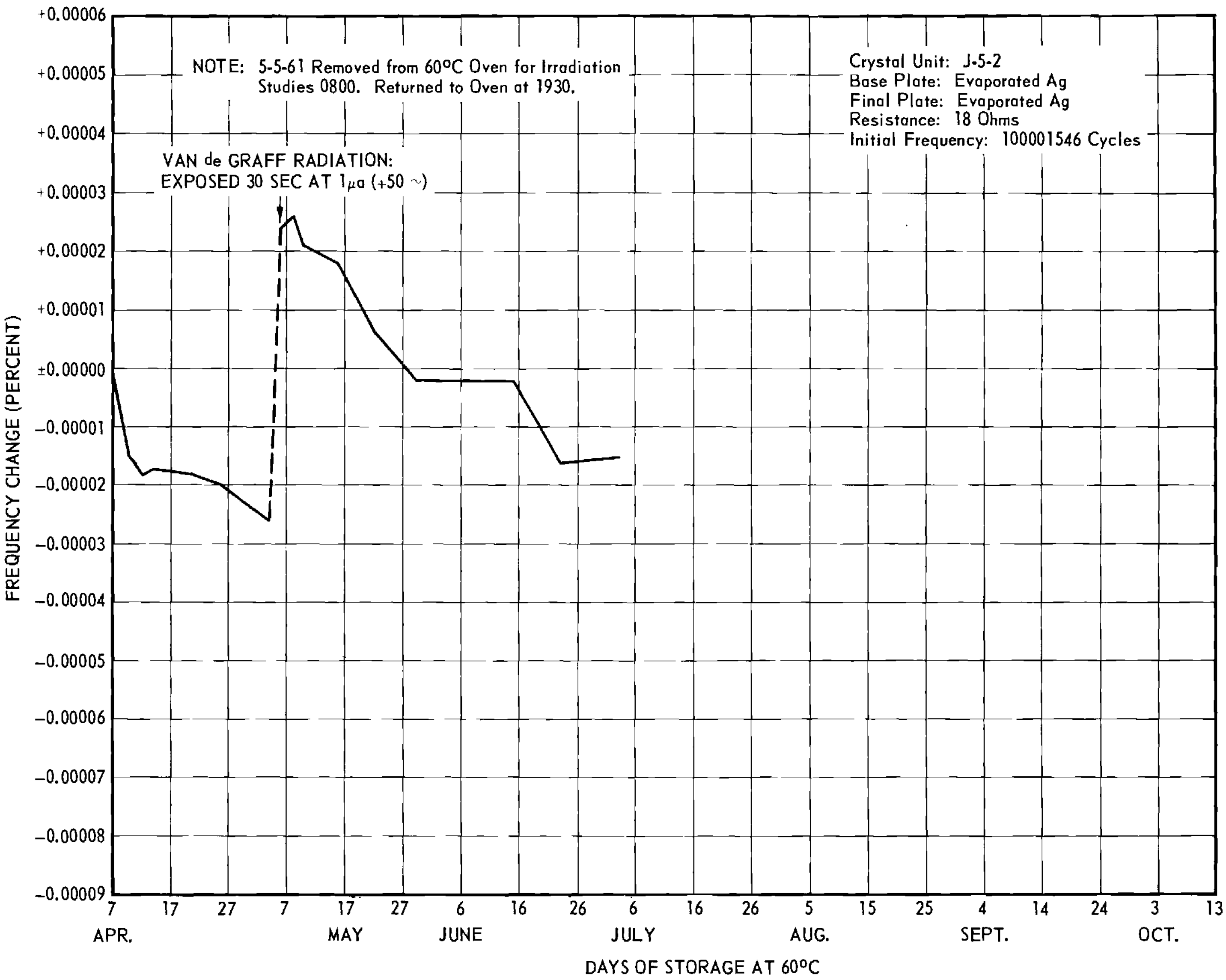

Figure 23. Frequency versus time data for resonator J-5-2-Ag+Ag, a fifth overtone unit plated with evaporated silver plus silver and stored at 60°C.

Resonators: K-5-1 to K-5-10

Blanks: 0.450" diameter, polished, etched, 20.200 Mc

Base Plate: 1500 A evaporated silver at 300°C substrate temperature

Final Plate: None

Mounting: W.R.W. stems with 0.006" spring clips

Bonding: duPont 5504 A cement cured three hours at 150°C

Vacuum Baking: 175°C for three hours

Comments: Yield: 80%. First group bonded with 5504 A epoxy-silver cement. These units showed characteristic and similar aging behavior. This was signified by a drop of 0.2 ppm in 30 days followed by leveling off at a slope of about 0.1 ppm per year.

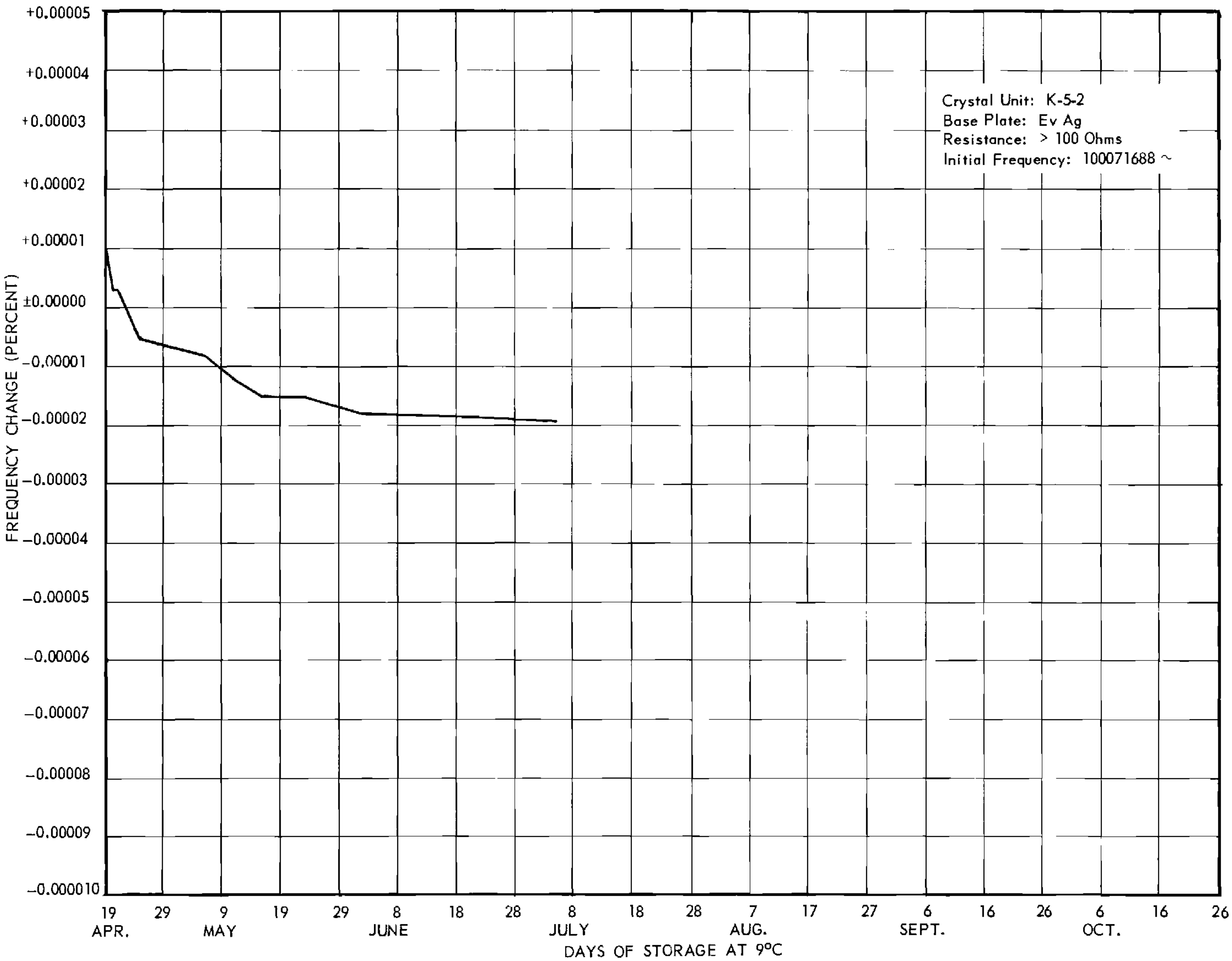

Figure 24. Frequency versus time data for resonator K-5-2-Ag, a fifth overtone unit plated with evaporated silver and stored at 0°C.

Resonators: A-7-1 to A-7-10

Blanks:	0.375" diameter, polished, etched, 14.370 Mc
Base Plate:	1500 A evaporated aluminum at 250°C substrate temperature
Final Plate:	None
Mounting:	G.E. stems with tab clips
Bonding:	Hanovia No. 2 cement
Vacuum Baking:	150°C for six hours
Comments:	Yield: 30%. Average resistance of operable units: 52 ohms

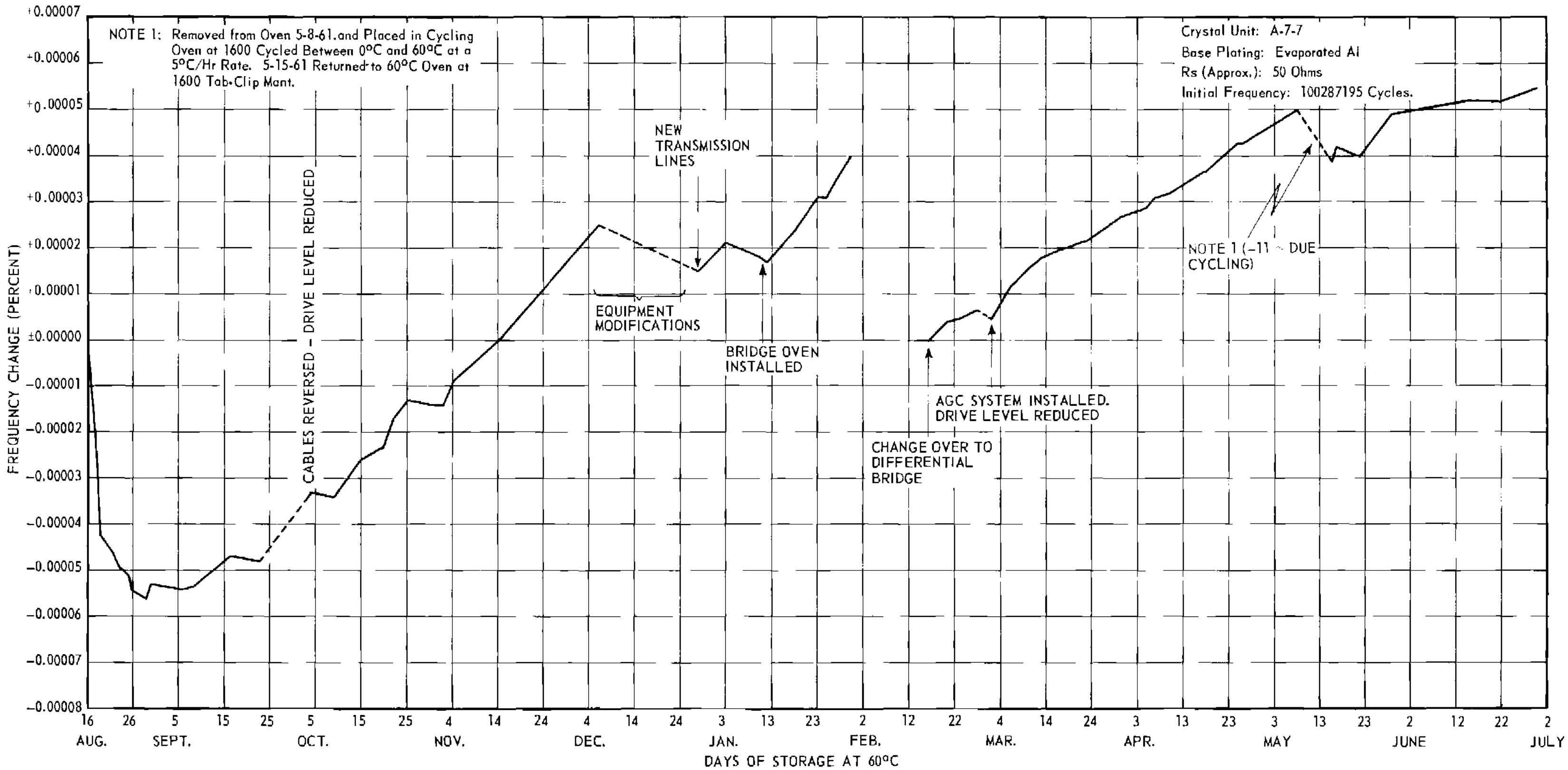

Figure 25. Frequency versus time data for resonator A-7-7-Al, a seventh overtone unit plated with evaporated aluminum and stored at 60°C.

Resonators: B-7-1 to B-7-10

Blanks: 0.450" diameter, polished, etched, 14.480 Mc

Base Plating: 1500 A evaporated silver at 300°C substrate temperature

Final Plating: None

Mounting: W.R.W. stems with 0.006" spring clips

Bonding: duPont 5504 A cement cured three hours at 150°C

Vacuum Baking: 175°C for three hours

Comments: Yield: 100%. Many of these units showed unaccountable positive shifts of +0.5 ppm before leveling off. Better units did not show positive shift and leveled off at about -0.2 ppm.

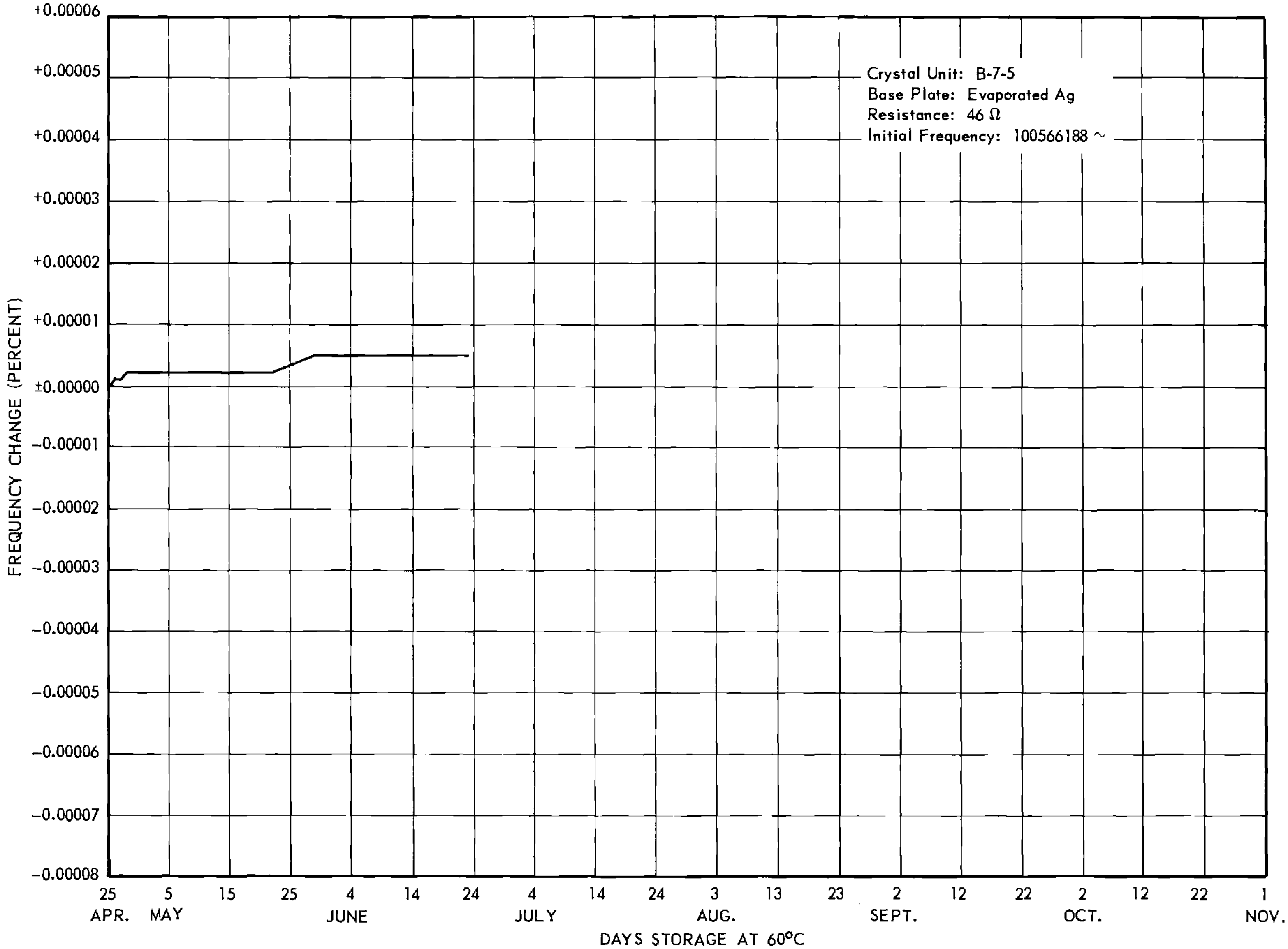

Figure 26. Frequency versus time data for resonator B-7-5-Ag, a seventh overtone unit plated with evaporated silver and stored at 60°C.

Resonators: C-7-1 to C-7-10

Blanks: 0.450" diameter, polished, etched, 14.480 Mc

Base Plating: 1500 A evaporated silver at 300°C substrate temperature

Final Plating: Evaporate silver. Average plate back: 400 kc

Mounting: W.R.W. stems with 0.006" spring clips

Bonding: duPont 5504 A cement cured three hours at 150°C

Vacuum Baking: 175°C for three hours

Comments: Yield: 70%. Units show erratic positive and negative shifts in most instances. A large part of this appears to be related to overheating during sealing.

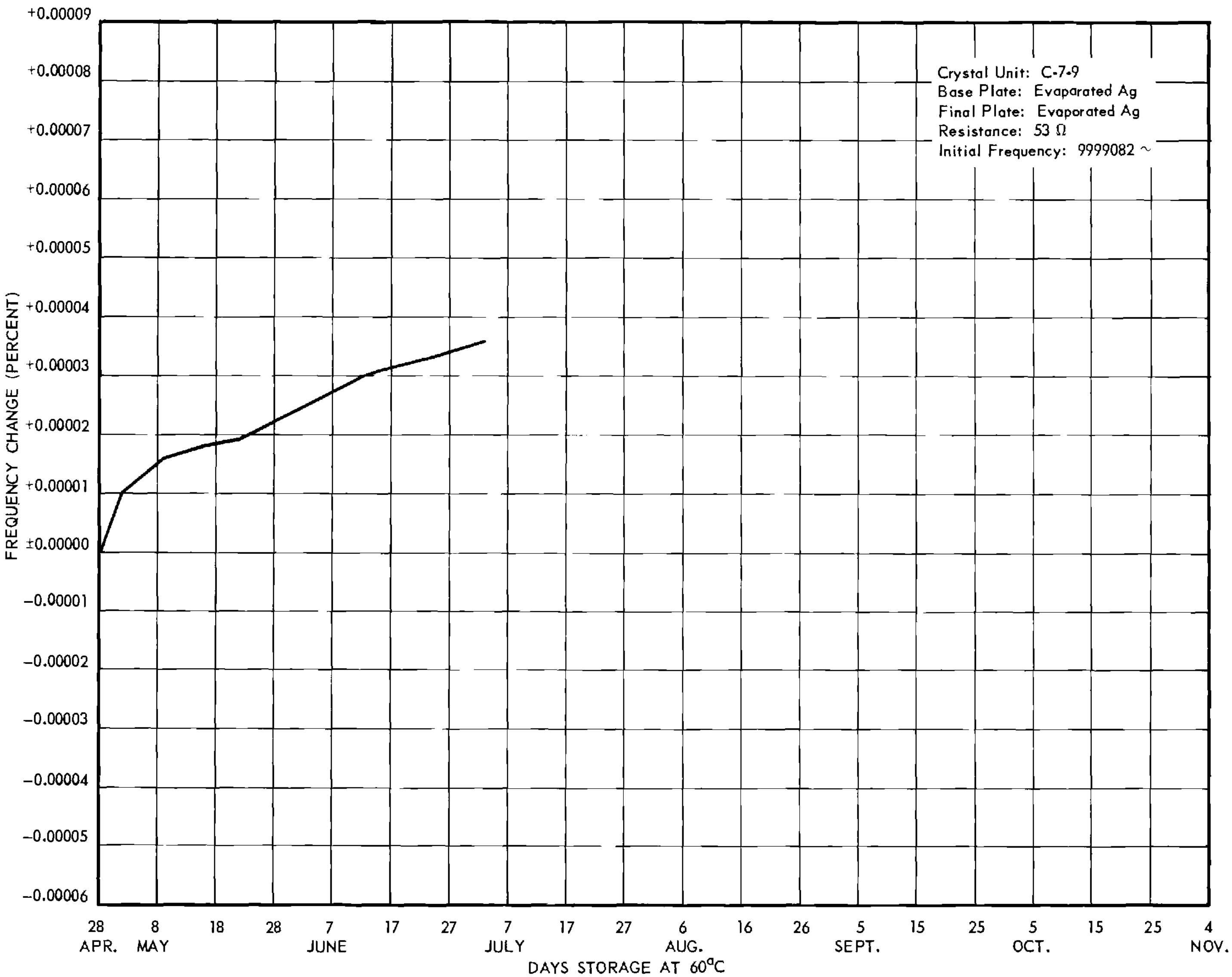

Figure 27. Frequency versus time data for resonator C-7-9-Ag+Ag, a seventh overtone unit plated with evaporated silver plus silver and stored at 60°C.

Resonators: A-9-1 to A-9-10

Blanks: 0.375" diameter, polished, etched, 11.200 Mc

Base Plating: 1500 A evaporated aluminum at 250°C substrate temperature

Final Plating: None

Mounting: G.E. stems with tab clips

Bonding: Hanovia No. 2 cement

Vacuum Baking: 150°C for six hours

Comments: Yield: 90%. The better units of this group exhibited very stable behavior before exposure to irradiation. However, the units exhibited shifts ascribable to instrumentation changes during the early measurement period and these confused interpretation of early data.

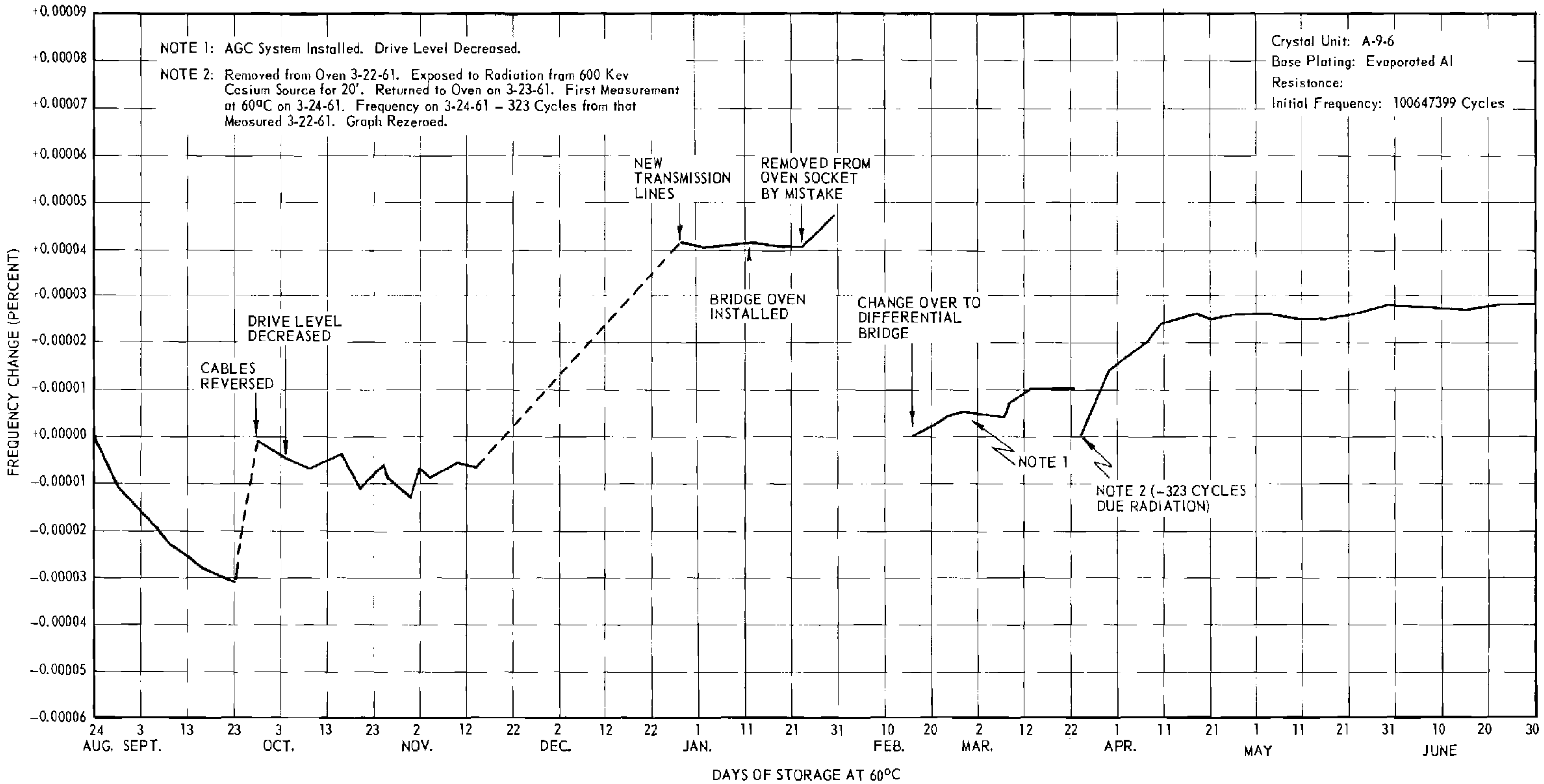

Figure 28. Frequency versus time data for resonator A-9-6-Al, a ninth overtone unit plated with evaporated aluminum and stored at 60°C.

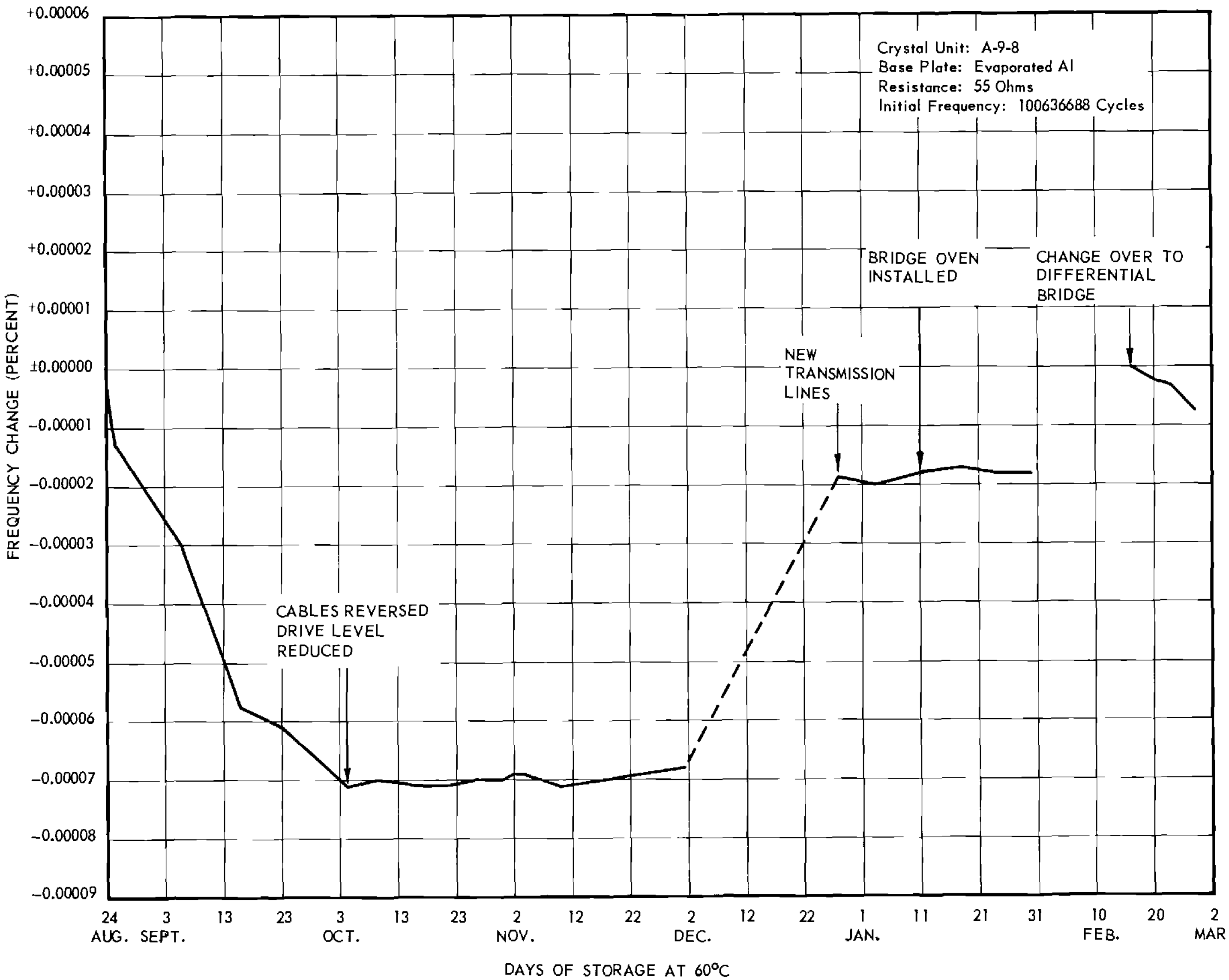

Figure 29. Frequency versus time data for resonator A-9-8-Al, a ninth overtone unit plated with evaporated aluminum and stored at 60°C.

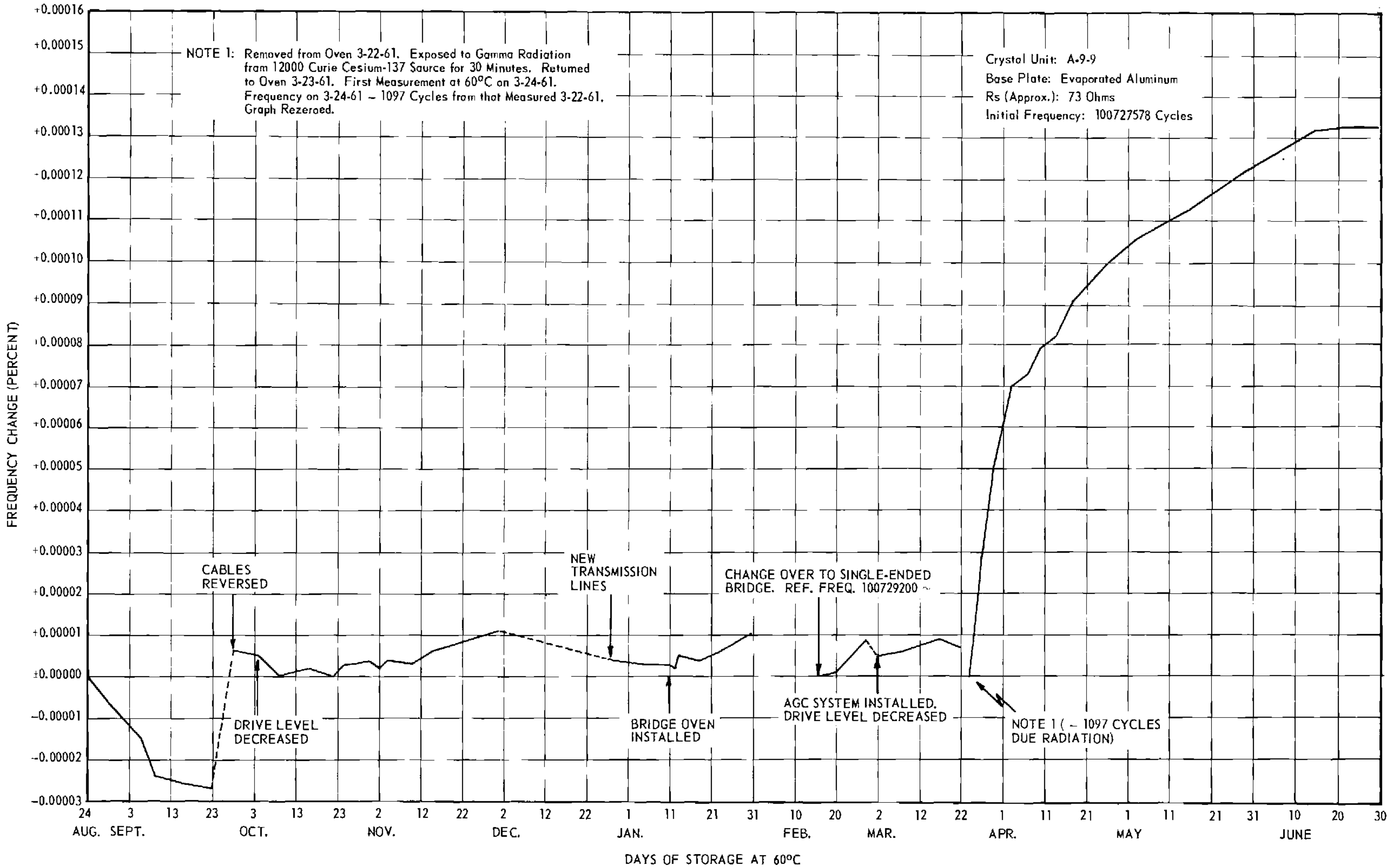

Figure 30. Frequency versus time data for resonator A-9-9-Al, a ninth overtone unit plated with evaporated aluminum and stored at 60°C.

Resonators: B-9-1 to B-9-10

Blanks: 0.460" diameter, polished, etched, 11.310 Mc

Base Plating: 1500 A evaporated silver at 250°C substrate temperature

Final Plating: None

Mounting: G.E. stems with tab clips

Bonding: Hanovia No. 2 cement

Vacuum Baking: 200°C for 5.5 hours

Comments: Yield: 50%. The surviving units were quite stable, holding ±0.2 ppm for a period of six months prior to irradiation studies.

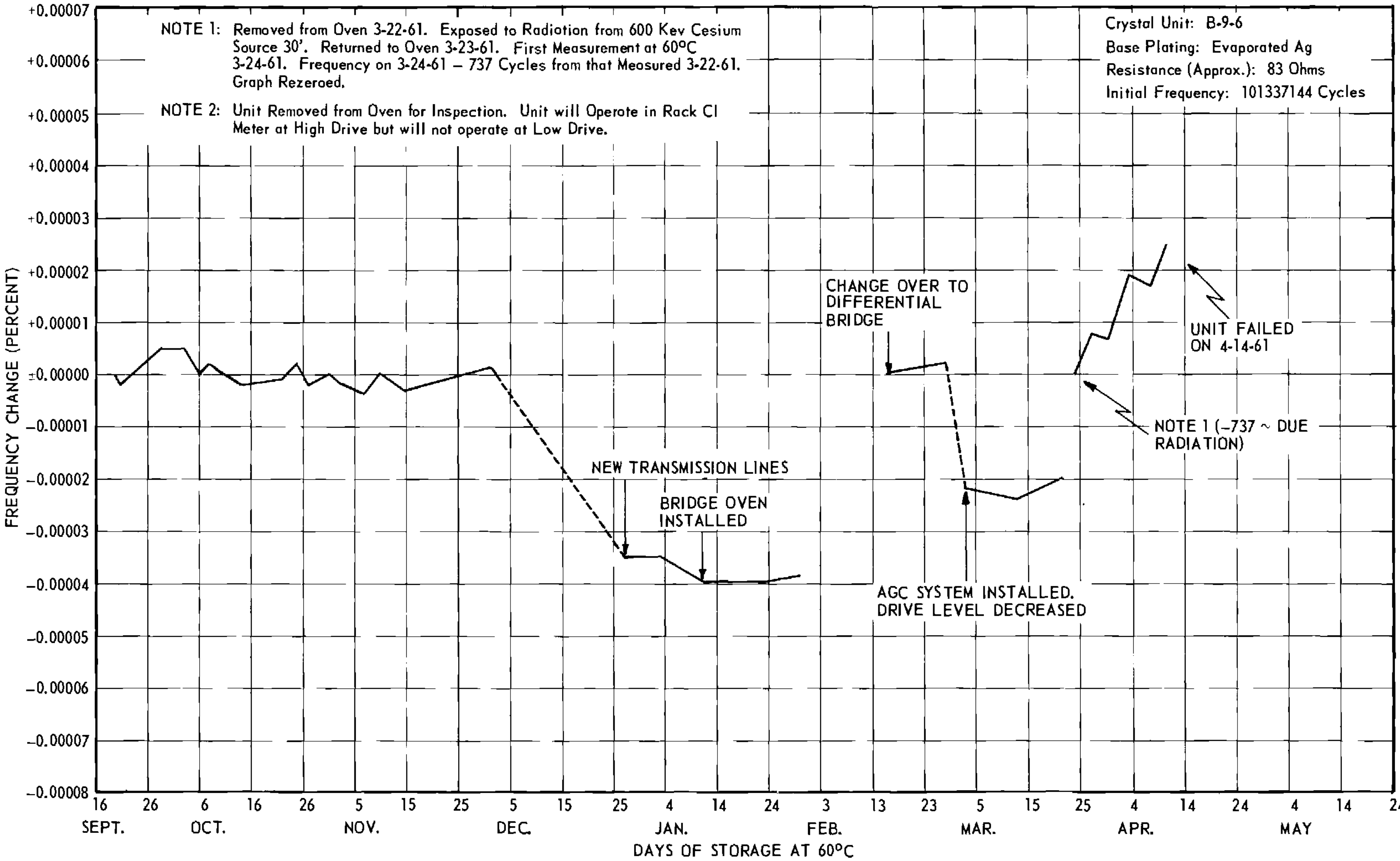

Figure 31. Frequency versus time data for resonator B-9-6-Ag, a ninth overtone unit plated with evaporated silver and stored at 60°C.

Resonators: C-9-1 to C-9-10

Blanks: 0.460" diameter, polished, etched, 11.310 Mc

Base Plating: 1500 A evaporated silver at 250°C substrate temperature

Final Plating: None

Mounting: W.R.W. stems with tab clips

Bonding: Hanovia No. 2 cement

Vacuum Baking: 200°C for six hours

Comments: Yield: 10%. Due primarily to leaking stems and stem-bulb seals. The one unit sealed performed quite well.

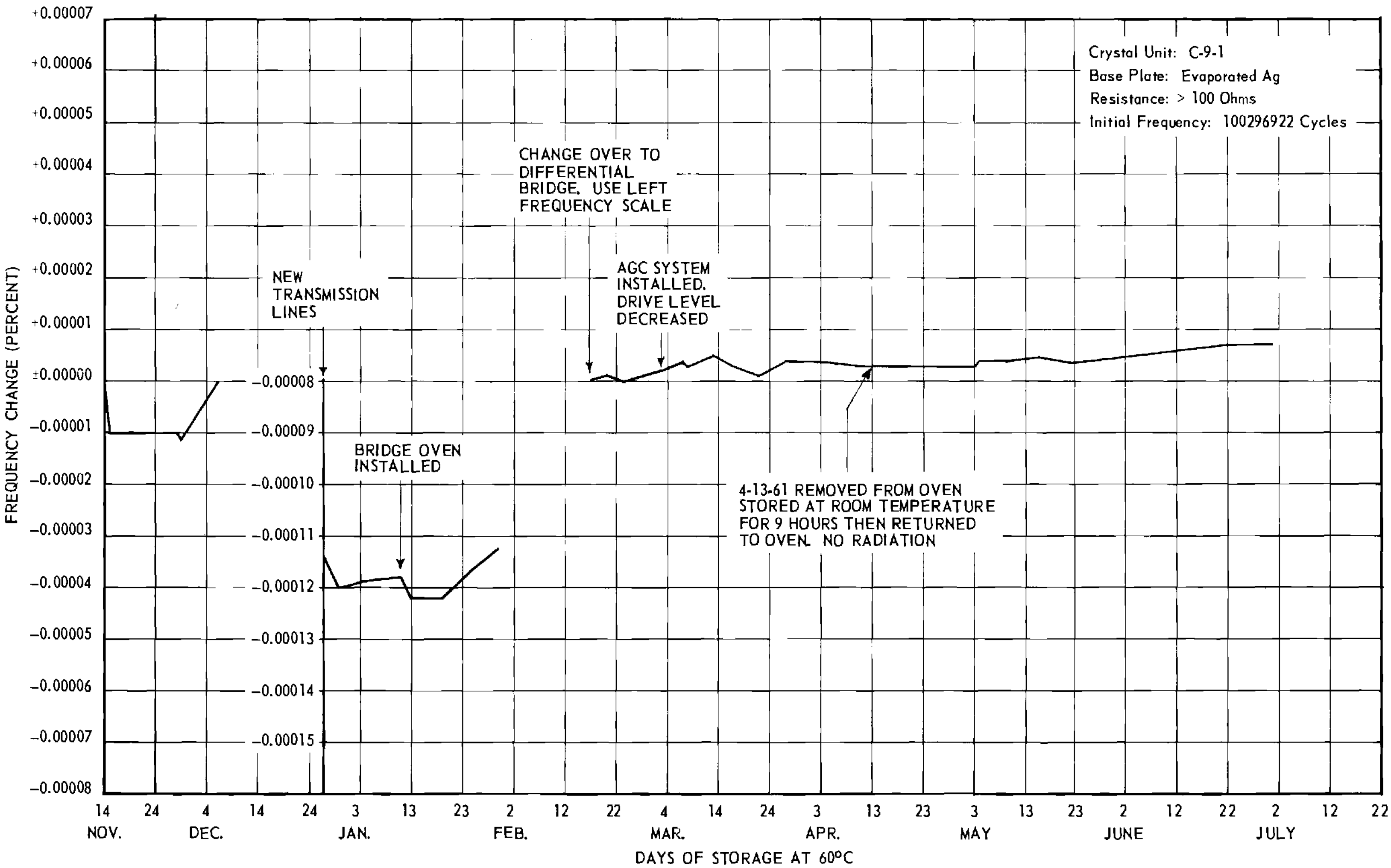

Figure 32. Frequency versus time data for resonator C-9-1-Ag, a ninth overtone unit plated with evaporated silver and stored at 60°C.

Resonators: D-9-1 to D-9-10

Blanks: 0.460" Diameter, polished, etched, 11.310 Mc

Base Plating: 1500 A evaporated silver at 150°C substrate temperature

Final Plating: None

Mounting: W.R.W. stems with 0.006" spring clips

Bonding: Hanovia No. 2 cement

Vacuum Baking: 200°C for six hours

Comments: Yield: 50%. These units performed stably after instrumentation problems were corrected.

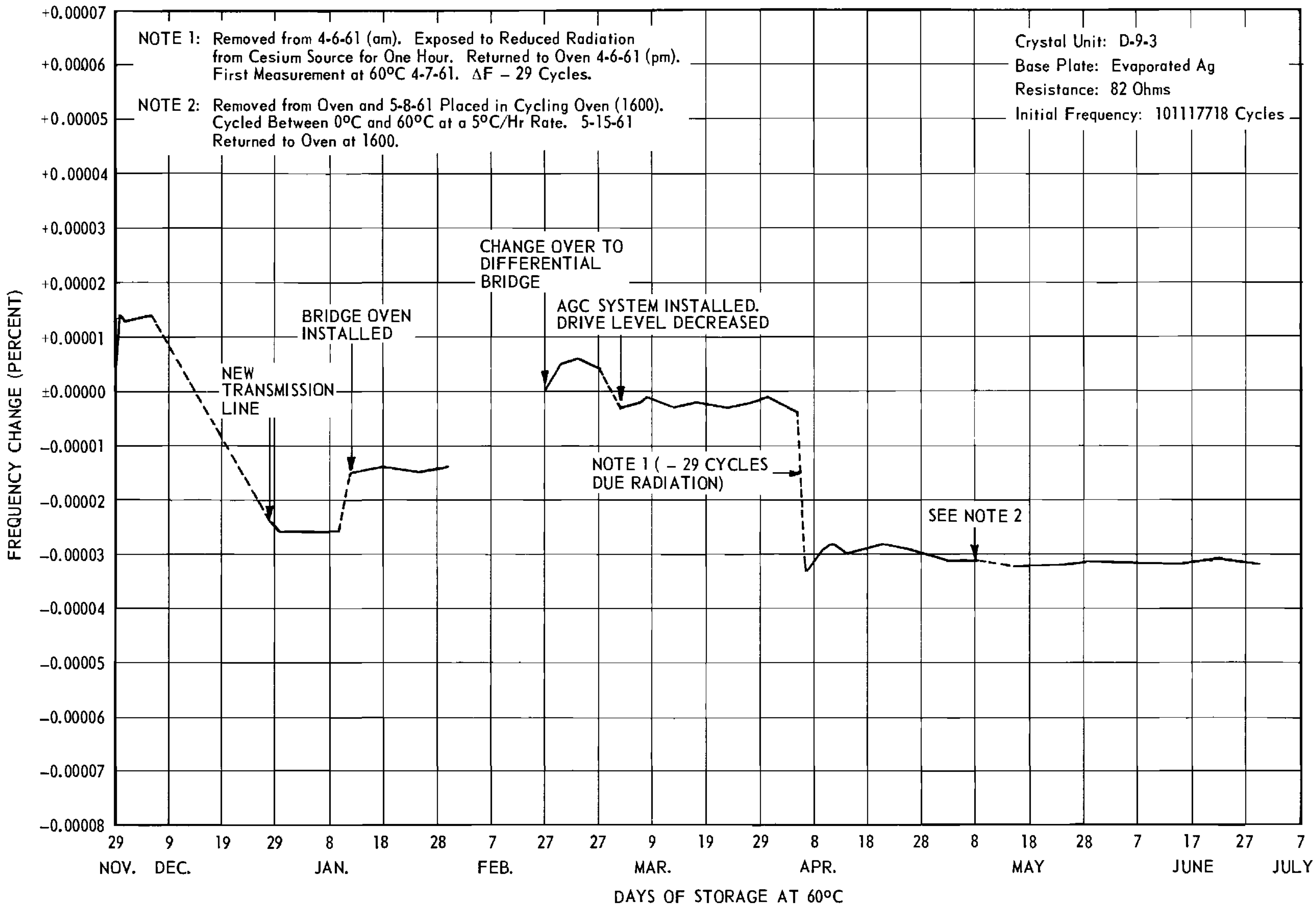

Figure 33. Frequency versus time data for resonator D-9-3-Ag, a ninth overtone unit plated with evaporated silver and stored at 60°C.

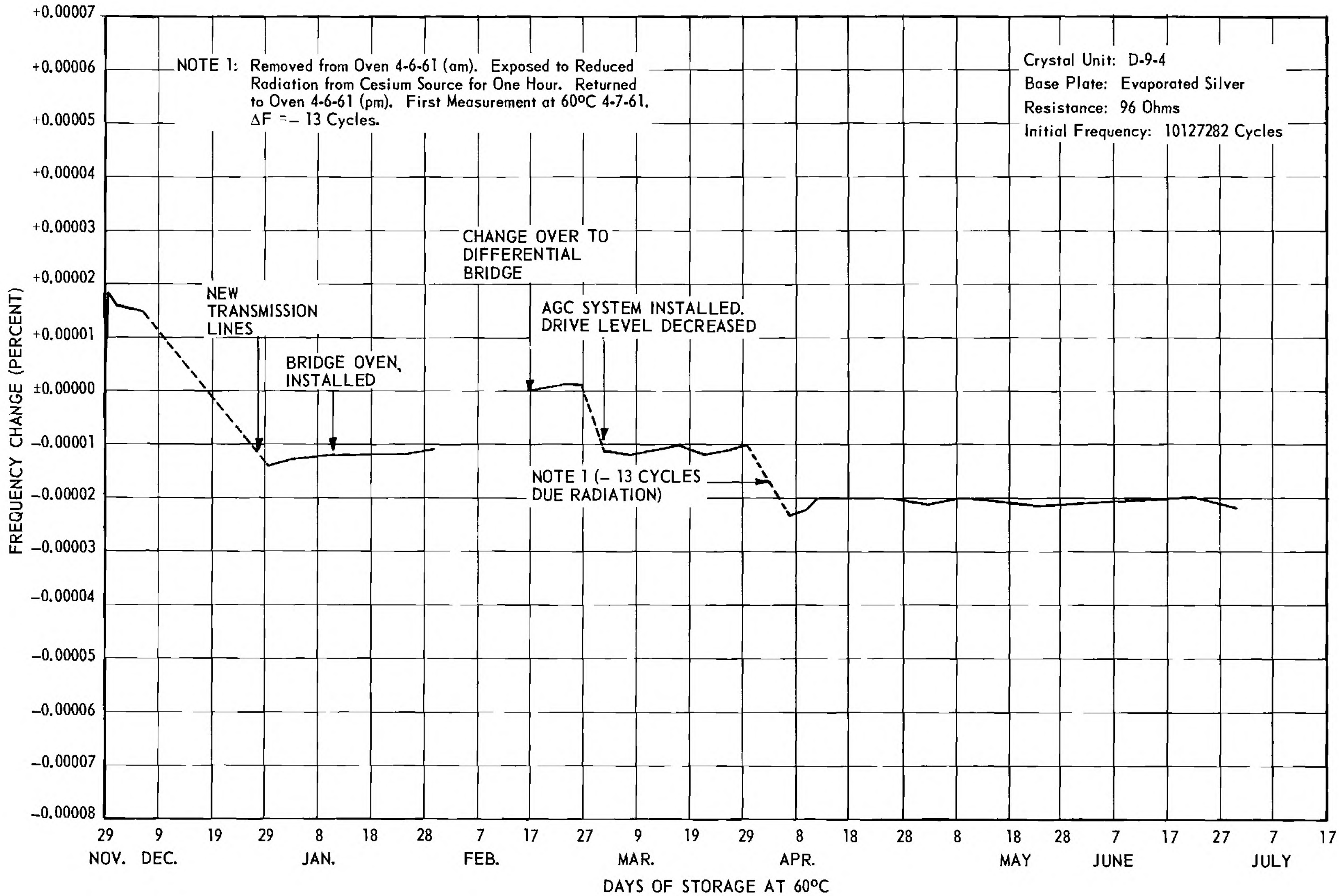

Figure 34. Frequency versus time data for resonator D-9-4-Ag, a ninth overtone unit plated with evaporated silver and stored at 60°C.

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